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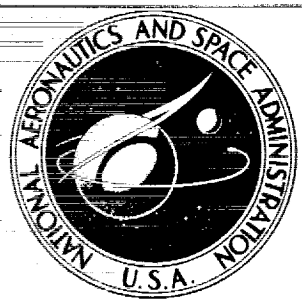

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DEVELOPMENT OF AN IMPROVED COATING FOR POLYBENZIMIDAZOLE FOAM

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Prepared by
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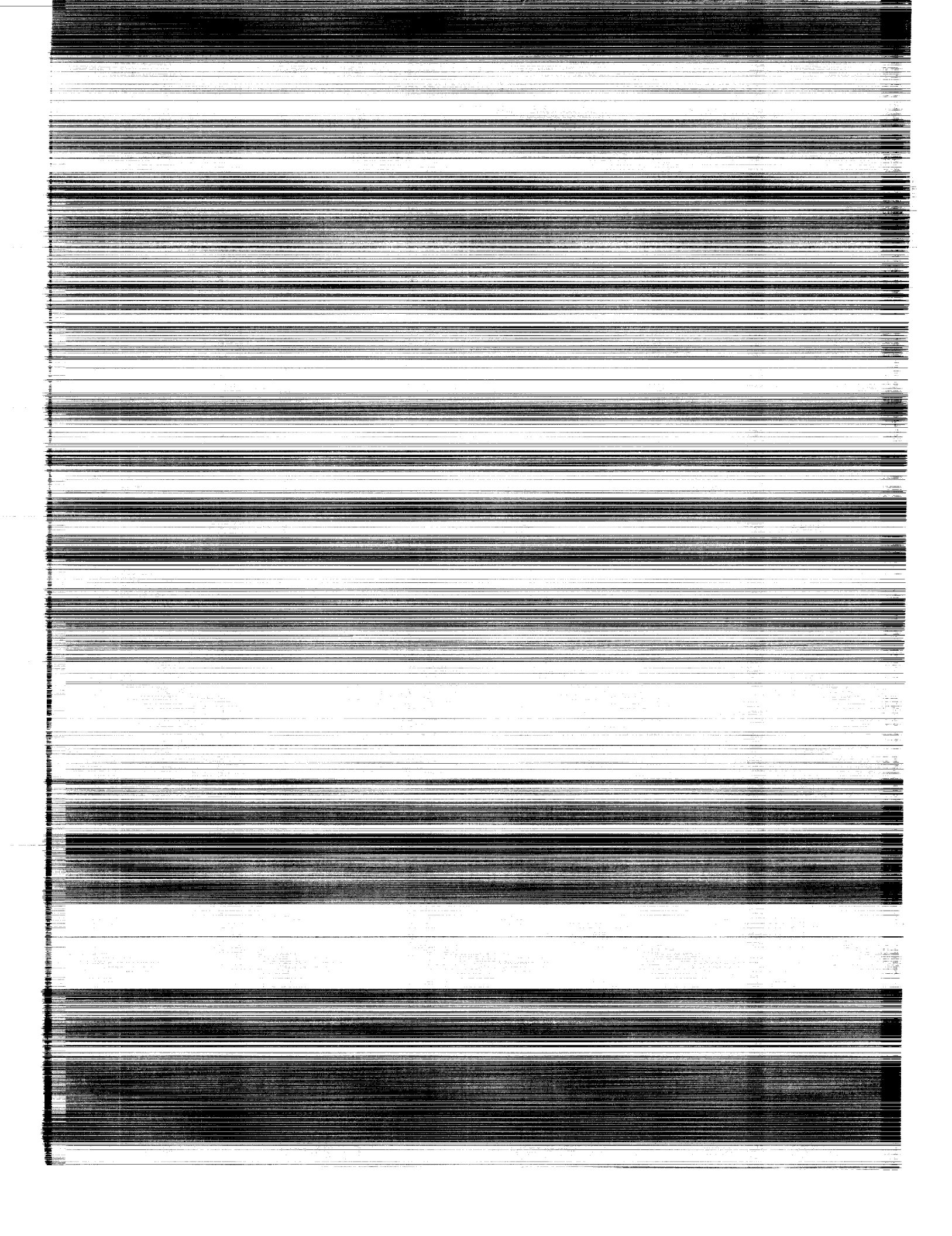

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16. Abstract

An improved coating system was developed for Polybenzimidazole (PBI) foam to provide coating stability, ruggedness, moisture resistance, and to satisfy optical property requirements ($\alpha_s/\varepsilon \leq 0.4$ and $\varepsilon > 0.8$) for the space shuttle. The effort was performed in five tasks: Task 1 to establish material and process specifications for the PBI foam, and material specifications for the coatings; Task 2 to identify and evaluate promising coatings; Task 3 to establish mechanical and thermophysical properties of the tile components; Task 4 to determine by systems analysis the potential weight trade-offs associated with a coated PBI TPS; and Task 5 to establish a preliminary quality assurance program.

The coated PBI tile was, through screening tests, determined to satisfy the design objectives with a reduced system weight over the baseline shuttle silica LRSI TPS. The developed tile provides a thermally stable, extremely rugged, low thermal conductivity insulator with a well characterized optical coating.

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FOREWORD

This final report was prepared by the Acurex Corporation, Aerotherm Division, and Whittaker Research and Development for NASA/Ames Contract NAS2-8490, Development of an Improved Coating for Polybenzimidazole Foam. This work was performed under the direction of the Thermal Protection Branch with Mr. Howard Goldstein as the Technical Monitor.

This report is arranged in accordance with the five major tasks performed under this contract during the period 8 August 1974 through 15 September 1975.

ABSTRACT

An improved coating system was developed for Polybenzimidazole (PBI) foam to provide coating stability, ruggedness, moisture resistance, and to satisfy optical property requirements ($\alpha_{s/\epsilon} \leq 0.4$ and $\epsilon > 0.8$) for the space shuttle. The effort was performed in five tasks: Task 1 to establish material and process specifications for the PBI foam, and material specifications for the coatings; Task 2 to identify and evaluate promising coatings; Task 3 to establish mechanical and thermophysical properties of the tile components; Task 4 to determine by systems analysis the potential weight trade-offs associated with a coated PBI TPS; and Task 5 to establish a preliminary quality assurance program.

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SECTION 1

SUMMARY

With the development of advanced reusable heat shield materials for space shuttle vehicle (SSV) applications, it became apparent that the lower surface heat shield material might not provide optimized thermal protection for the upper surface areas where the environments are much less severe. Polybenzimidazole (PBI) foam tiles were evaluated as an alternate to the silica reusable surface insulation (RSI) currently baselined for the SSV lower surface. Coated PBI foam was proposed due to the following potential benefits:

- Low weight
- Extended low and high temperature capability 117°K to 728°K (-250°F to 850°F)
- Low maintenance
- Compatibility with structure
- Ruggedness

The material had been tested to determine its basic properties and response to both radiant lamp and arc plasma simulated entry environments. Test samples of a coated PBI foam system were supplied to both NASA and Rockwell International for evaluation. Results of these evaluations demonstrated a weight advantage for the PBI foam system as compared to the silica RSI type of insulator. PBI foam appeared to be a more compliant and tough TPS system.

These preliminary evaluations, however, exhibited certain deficiencies which appeared correctable without a performance penalty:

- Coating mechanical and thermal stability
- Long term oxidation resistance

This program was initiated to address these problems.

The primary objective of this program was to develop a coating system for PBI foam that would

- Provide necessary optical properties
- Retain properties for the equivalent of 100 space shuttle flights
- Provide a moisture seal
- Provide oxidation protection

In addition, the feasibility of direct bonding the PBI foam to an aluminum structure was to be demonstrated.

The program consisted of five tasks:

- Task 1 Establish material and process specifications for the PBI foam and material specifications for the coatings.
- Task 2 Identify and evaluate promising coatings
- Task 3 Establish mechanical and thermophysical properties not currently available.
- Task 4 Determine by systems analysis the potential of coated PBI TPS.
- Task 5 Establish a preliminary quality assurance program.

The objective of Task 1 was to generate the material and processing specification for the PBI foam and the material specifications of the thermal control coating. The PBI foam material and processing specifications characterize material screening and processing techniques including raw material requirements, prepolymer quality control and packaging requirements, foam preparation, and foam quality control. The thermal control coating material specifications characterize the VHT SP101 coating selected in this program.

The objective of Task 2 was to investigate materials, tile design concepts, and tile processing techniques required to develop an insulation system based upon PBI foam which would provide thermal protection in environments typical of the space shuttle upper surface. In this task candidate packaging and thermal control coatings were selected, fabricated, and evaluated. The screening tests performed included foam moisture tests, shear tests, thermal shock tests, impact tests, optical property tests, outgassing tests, and porosity tests. This task identified VHT SP101 and glass cloth layups as the optimum

coating system from both a stability and an optical property standpoint. The feasibility of directly bonding PBI tiles to an aluminum structure was successfully demonstrated.

The objective of Task 3 was to establish the mechanical and thermophysical properties required to fully characterize the PBI system. Each component of the PBI tile was tested for tensile strength and thermal expansion. Also, the thermal conductivity of the PBI foam was evaluated. These data, along with data obtained previously, provided the required data base for analyzing the performance of the coated PBI foam in projected operational environments (e.g., shuttle, hypersonic aircraft, space tug, etc.).

The objective of Task 4 was to perform sensitivity analyses, thermal sizing and structural evaluation of the PBI TPS to identify the optimum tile construction, attachment techniques, and size. The effects of optical properties, bondline thickness, and foam density upon the TPS thermal performance were evaluated. Structural evaluations identified both the critical structure components of the time and the structural limitations upon tile size in the shuttle operational environment. In addition, PBI TPS sizing identified the potential weight savings over the baseline shuttle LRSI TPS.

The objective of Task 5 was to establish a minimum quality assurance program to insure NASA that continuity could be maintained if the need should arise to scale-up the PBI development to a full scale production effort. The purpose of this effort was to inspect all documentation, identify and evaluate quality problems, and to maintain overall liaison with WR&D QA personnel.

The contract objectives were met, and the viability of the PBI TPS for typical shuttle operational environments was demonstrated. Additional radiant lamp testing is currently being performed to verify the thermal performance of the selected tile concept.

SECTION 2

TECHNICAL DISCUSSION OF TASKS

2.1 INTRODUCTION

A critical component of the space shuttle vehicle (SSV) presently under development by NASA is the reusable surface insulation (RSI) heat shield. The baseline system for both the lower and upper surfaces of the vehicle is LI-900, a silica RSI. It has been estimated that polybenzimidazole (PBI) foam offers a significant saving in heat shield weight over silica low temperature RSI baselined for the upper surface of the shuttle. PBI foam offers low density $\leq$ 64 kg/m^3 ($\leq$ 4 lb/ft^3) with excellent thermal stability and can be directly bonded to the structure eliminating the strain isolation pad required by the silica RSI system. In addition, the technology developed in this program is applicable to other thermal protection requirements within the operational thermal limitation of the PBI tile. Such potential applications include space tug, hypersonic aircraft, etc..

This program to develop an improved coating system for the PBI foam was initiated on 8 August 1974. The program consisted of five tasks and the results of these tasks are summarized in the following sections.

2.2 TASK 1 — MATERIAL AND PROCESSING SPECIFICATIONS

At the outset of this program, PBI foam material and processing specifications were developed for the nominal 64 kg/m^3 (4 lb/ft^3) foam from previous Whittaker R&D efforts. Throughout the program, modifications were incorporated into the specifications to more completely characterize raw material screening and processing techniques. The resultant material and processing specifications for the PBI foam are presented in Appendix A; these specifications include:

- Description of raw material requirements
- Prepolymer quality control requirements
- Prepolymer packaging requirements

- Foam preparation including
  - Quantities of prepolymer required
  - Facilities and tooling
  - Processing techniques
- Foam quality control

In addition to the foam specifications, material specifications for the baseline thermal control coating (VHT SP101) were formulated. These specifications are presented in Appendix B.

2.3 TASK 2 – COATING TECHNIQUE EVALUATION

The objective of this task was to investigate materials, tile design concepts and processing techniques required to develop an insulation system based on PBI foam which will provide thermal protection in environments typical of the shuttle upper surface. Shuttle, of course, is only one of the several vehicles which could benefit from success on this program. A smooth flight surface and precise geometry are required for both engineering design and installation. Accordingly, a title concept evolved to solve these problems.

An additional objective was to develop a thermal control coating system for the PBI foam which would provide adequate α/ε values [$(\alpha/\varepsilon)_{RT} \leq 0.4$ and $\varepsilon_{T_{max}} \geq 0.8$] and remain unchanged in terms of optical properties for the equivalent of 100 space shuttle flights. The coating had to be stable in hard vacuum under solar radiation for the equivalent of 300 days and provide, if possible, an oxidation barrier for the foam.

In order to satisfy these task objectives, a matrix of tile fabrication methods and optical control coating materials and applications were investigated. Section 2.3.1 reviews these candidate tile fabrication techniques and the optical control coatings evaluated throughout the program. Subsequently, Section 2.3.2 reviews both the results of the concept fabrication evaluations summarized in Section 2.3.1 and the selection of the baseline tile fabrication technique. Tile fabrication experience gained in the fabrication of radiant lamp test specimens is also summarized in Section 2.3.2.

2.3.1 Candidate Selection

At the outset of the program, candidate tile concepts and optical control concepts were identified for investigation. Section 2.3.1.1 reviews the tile concepts examined, and Section 2.3.1.2 identifies the optical coatings evaluated.

2.3.1.1 Candidate PBI Tile Concepts

PBI tile packaging must provide dimensional stability, a coatable surface, and an oxidation barrier. In addition, PBI foam swells slightly after fabrication as it equilibrates with atmospheric moisture. Hence, when dry PBI foam is bonded to a sheeting material, the panel will be flat immediately after bonding. However, as the panel picks up moisture, it warps slightly. Previous experience has demonstrated that this warp can be eliminated if the panel is skinned on both sides. Consequently, various overlay/skin concepts have been investigated to:

- Provide a smooth, continuous bondable surface which acts as a substrate for the thermal control coating.
- Provide the underlying foam with an additional barrier to oxidative attack.
- Provide an additional barrier to water absorption by the foam.
- Provide a tough surface which will resist mechanical damage during handling and erosion of the face and edge of the tile during flight.

An optimum overlay should accomplish these functions, be cost effective, and have miminum weight.

Earlier experimental work utilized a thin metallic foil (e.g., aluminum, nickel, stainless steel) bonded directly to the surface of the foam. Several deficiencies were evident with metal foil overlays:

- Gross surface imperfections on the foam were translated to the metal foil surface.
- Bonding of the foam to the metal foil with PBI resin was unsatisfactory.

The metal foil overlay concept was abandoned.

In this task of the program, three candidate concepts were investigated.

- No overlay
- Overlay as top skin
- Overlay over skinned foam

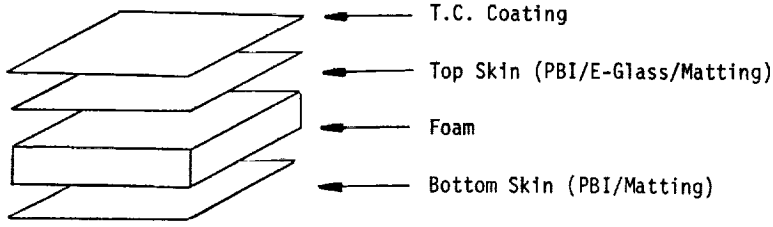
Candidate materials for the overlays and skins include:

- Quartz Fabric
- E-Glass Fabric
- E-Glass Matting

Previous tile fabrications had used quartz fabric as an overlay material. However, significantly reduced tile exposure temperature design constraints (728°K instead of 811°K) led to the investigation of using E-glass materials for skins and overlays. In addition, E-glass materials are currently available at a lower cost and in lower weight fabrics than commercially available quartz fabrics. Various combinations of glass cloth and matting were evaluated for skin/overlay layups to optimize surface skin flatness, porosity, and weight. The basic fabrication techniques investigated are reviewed below.

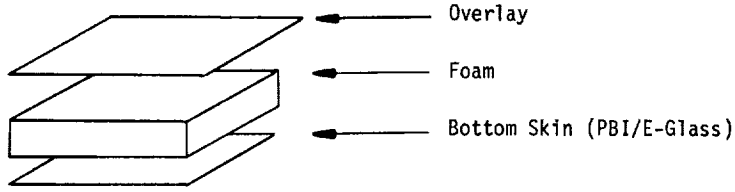
No Overlay

In this concept the thermal control coating is applied directly to the skinned foam. For example, VHT SP101 is sprayed onto a layer of PBI/E-Glass fabric. The weight of the overlay is thereby eliminated. Previous studies have demonstrated that coatings, such as Z-93, adhered quite well to PBI/quartz or to a PBI/glass balloon syntactic.



Overlay as Top Skin

In this concept the foam has a bottom skin of PBI E-Glass fabric and the top foam surface bonded directly to the overlay. The overlay functions as the top skin to prevent tile warpage:



Candidate overlay materials are listed below:

Matrix Systems:

1. Slip-cast ceramics such as SiO_2, BN, $AlMgO_x$, $MgSiO_x$ and Al_2O_3
2. $AlPO_4$ with fillers to tailor the expansion coefficient
3. PBI resin

Reinforcements/Fillers:

1. Fiberfrax paper
2. Quartz and glass fabrics
3. Quartz and glass matting
4. Hollow glass beads

Flame Sprayed Refractories

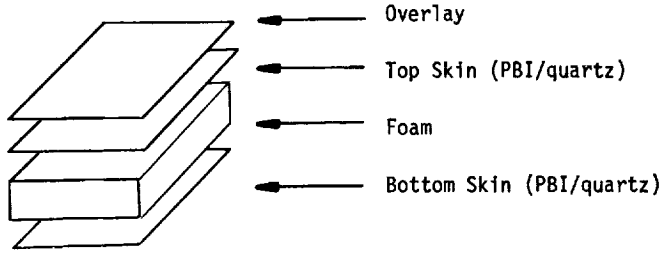
1. Al_2O_3
2. Cr_2O_3
3. Zr_2O_3

The different reinforcements and matrix materials could be combined, either separately or in situ, to form a coherent, thin layer.

The most critical parameter to be evaluated with this concept was the adhesion of the overlay to the foam. If a secondary bonding operation were utilized, the adhesive could be organic (PBI) or inorganic (e.g., the matrix of the overlay). If cured in situ the adhesive would be the excess matrix from the overlay.

Overlay Over Skinned Foam

In this concept a top and bottom skin was to be applied to the low density foam prior to the application of the overlay to the top skin:



Materials for the overlay in this concept are as listed in the preceeding paragraph. The most important property to be investigated here was the adhesion of the overlay to the skin.

Reference System

The last concept listed above was in use at the beginning of the program and it is depicted in Figure 2-1. It required a multistep fabrication using quartz fabric, PBI, Chemceram and Z-93 in its construction. Briefly the process required the following steps:

1. Impregnate two sheets of quartz fabric with PBI and cure into skins with a final cure cycle of 1 hour at 672°K (750°F).
2. Apply PBI adhesive to one side of each skin.
3. Cure skins to PBI foam with a final cure of 1/2 hour at 769°K (925°F).
4. Make a quartz fabric reinforced Chemceram hat (geometry shown in Figure 2-1).
5. Apply Chemceram adhesive to the inside of hat and bond to PBI quartz foam sandwich described above.
6. Send to subcontractor for application of Z-93 thermal control coating.

2.3.1.2 Candidate Thermal Control Coating Selection

The purpose of the thermal control coating is to provide the correct ratio of absorbed (α_s) to emitted (ϵ) radiant energy throughout the operational thermal envelope typical of the upper surface of the space shuttle. A maximum value of $\alpha_s/\epsilon)_{RT} \leq 0.4$ was targeted with $\epsilon \geq 0.8$. The coating must also be stable in hard vacuum under solar radiation for the equivalent of 300 days. This coating should, in addition, exhibit properties consistent with ground handling and storage environment requirements. Highly desirable would be a coating which could be cleaned between flights in case of accident or contamination.

A literature review of thermal control coatings which might be candidates for the PBI LRSI tiles revealed the following state of the art conditions:

1. Testing of previous α_s/ϵ coatings had not in general been carried out to the target temperatures of the present program.

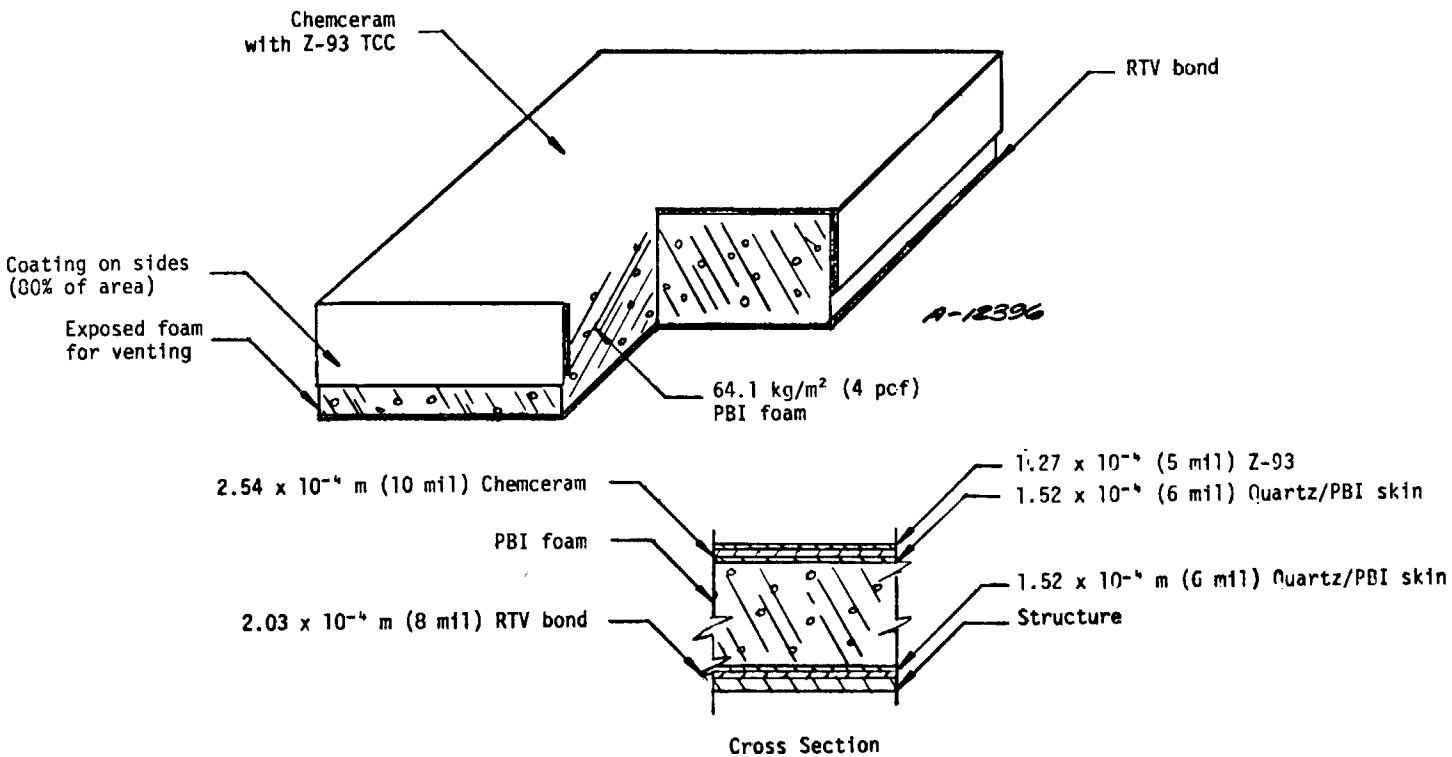


Figure 2-1. Reference PBI LRSI TPS tile concept.

2. When elevated temperatures were investigated, they were for single cycle α_s/ε measurements at elevated temperatures; not for changes in α_s/ε after cyclic exposure to high temperatures.

3. Pigment or coating degradation due to solar exposures exceeding 2000 equivalent sun hours had, in general, not been investigated.

4. Potential refurbishment methods were not documented.

5. Z-93 (silicated ZnO) thermal control coating is available and well documented.

In summary, very little work has been accomplished in developing thermal control coatings for use in both reentry and space environments.

The availability of space stable thermal control coatings which could be used in reentry heating environments appears very limited. However, there are a few inorganic and semi-inorganic materials systems which remain as candidates for this application.

Processing limitations imposed by the maximum reuse temperature of the PBI foam prohibit the direct application of coatings to the PBI foam when firing above 811°K (1000°F) is required. This limits the use of porcelain enamel or other high temperature glass type coatings. However, it is possible to apply such coatings independently to an overlay which in turn can be bonded (at lower temperatures) to the foam. The use of bonded glass fabrics or mats was the easiest way to achieve this goal as glass/binder materials can be selected with the correct α_s/ε properties and integrally or secondarily bonded to the foam.

Considering the optical coating design objectives, the candidate coatings selected for evaluation were:

- Flame Sprayed Coatings
  - Al_2O_3
  - Zr_2O_3
- Sprayable
  - VHT (SP 101): TiO_2 filled silicone
  - DC 805: TiO_2 filled silicone elastomer
  - Sauereisen Ceramics
- Overlay Concepts
  - E-glass fabric bonded with a ceramic cement

2.3.2 Fabrication Evaluations

The candidate tile fabrication techniques have been evaluated, and these fabrication experiences are summarized in Section 2.3.2.1. In addition, selection of a baseline fabrication technique and subsequent fabrication experience gained in the construction of radiant lamp test specimens are reviewed in this section. Coating fabrication technique evaluations are summarized in Section 2.3.2.2

2.3.2.1 Tile Fabrication Evaluation

A 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") tile geometry was selected as a baseline for evaluating fabricability of the candidate PBI tile packaging concepts. The objective of minimizing the tile weight dictated that the majority of the fabrication evaluation effort should be directed towards the no overlay concept.

2.3.2.1.1 Overlay Concept Evaluation

The first tile fabrication efforts evaluated the overlay fabrication technique. In this technique, precured skins are bonded directly to the tile. Various candidate sheeting materials were evaluated for use in the skin fabrications, and Table 2-1 presents a description of these fabrication results. All of the fabrics tested as overlays provided smooth tile surfaces (free from visual dimples), however it was observed that the surface porosity increased as the fabric weight was reduced. The results summarized in Table 2-1 indicate large variations in the adhesive weights [5.7×10^{-3} to 13.3×10^{-3} kg] (0.0126 to 0.0293 lbs). Thus, a need for controls over the adhesive application technique exists.

TABLE 2-1. OVERLAY CONCEPT FABRICATION EVALUATION*

Sheet Material	0.152m x 0.152m (6" x 6") Weight, kg x 10^3 (lb)	Impregnated, Cured with PBI, 0.152m x 0.152m (6" x 6"), grams		Weight of 2 PBI Skins, kg x 10^3 (lb)	PBI Foam (64 kg/m^3 nominal) 0.152m x 0.152m x 0.0127m (6" x 6" x 1/2") Tile Weight, kg x 10^3 (lb)	Weight of Cured Panel Skinned 2 Sides, kg x 10^3 (lb)	Weight of[a] Cured PBI Adhesive kg x 10^3 (lb)
		Weight, kg x 10^3 (lb)	Thickness, m (mils)				
E-GLASS							
104 Fabric	0.46 (0.0010)	0.93 (0.0020)	9.40×10^{-8} (0.0037)	1.86 (0.0041)	20.58 (0.0454)	30.94 (0.0682)	8.5 (0.0187)
112 Fabric	1.67 (0.0037)	2.87 (0.0063)	1.83×10^{-7} (0.0072)	5.74 (0.0126)	20.61 (0.0454)	39.69 (0.0875)	13.3 (0.0293)
116 Fabric	2.50 (0.0055)	3.74 (0.0082)	1.91×10^{-7} (0.0075)	7.48 (0.0165)	19.2 (0.0423)	37.66 (0.0830)	11.0 (0.0242)
181 Fabric	6.89 (0.0152)						
Surface Mat	0.69 (0.0015)	2.21 (0.0049)	1.91×10^{-7} (0.0075)	4.42 (0.0097)	18.9 (0.0417)	29.20 (0.0644)	5.9 (0.0130)
Swirled Mat	0.98 (0.0022)	2.14 (0.0047)	1.91×10^{-7} (0.0075)	4.28 (0.0094)	19.8 (0.0436)	31.17 (0.0687)	7.1 (0.0156)
QUARTZ							
503 Fabric (Reference)	2.86 (0.0043)	4.5 ± 1.5 (0.0099 ± 0.0033)		9.0 ± 0.3 (0.0198 ± 0.0066)	20 (assumed) (0.0441)	36.6[b] (0.0807)	7.6[c] (0.0168)
581 Fabric	6.84 (0.0151)						
Astro Mat	2.12 (0.0047)	6.73 (0.015)	3.68×10^{-7} (0.0145)	13.45 (0.0297)	20.58 (0.0454)	36.05 (0.0795)	2.0[d] (0.0044)

[a] Adhesive weights were obtained from finished tile weight, foam weight and the weight of two PBI skins
[b] Calculated
[c] Thinnest glueline successfully used in baseline tiles
[d] Suspect weighing error in data

* 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") PBI tiles with two layers of sheeting material (overlay concept)

2.3.2.1.2 No Overlay Concept Evaluation

In the no overlay concept, PBI prepolymer impregnated fabrics are cured directly to the foam. Thus, the adhesive layer required to bond the precured skins to the foam in the overlay fabrication method is eliminated. Tile weights can consequently be reduced.

Three panels were fabricated in this demonstration. The weight savings goal from the elimination of the PBI adhesive layer was verified for these tiles (summarized in Table 2-2) thus quantifying the advantage of the no overlay packaging concept. However, this effort demonstrated that dimples occurred over holes in the foam when fabrics were used but not when matting was used. Subsequent fabrication efforts using the no overlay technique therefore used matting in the skins to provide a smooth (free from visual dimples) coating substrate.

2.3.2.1.3 Evaluation of Overlay as Top Skin

An alternate method of packaging of the PBI foam is to precure a hat geometry skin, and then bond it to the PBI foam with an adhesive layer. After briefly investigating this concept, it was concluded that the cure shrinkage of the hat would require careful study. Whittaker Research and Development's 320. kg/m^3 (20 pcf) PBI syntactic foam was easily molded into a suitable hat geometry, however techniques to accomodate its 1 percent cure shrinkage were not developed. Increased weight due to the adhesive layer and the cure shrinkage problems associated with the hat made this an undesirable tile concept.

2.3.2.1.4 Baseline Concept Selection

The preliminary study on tile concepts resulted in the selection of a prime candidate to be further optimized. Initial screening tests indicated that both methods of bonding the skins to the foam are structurally acceptable; test specimens exhibited core failure in shear testing rather than skin delamination. Simplicity of fabrication and minimum weight were the prime factors in selecting the baseline system to be further optimized. This concept is to cure the PBI impregnated skins directly to the PBI foam. Two problems however had to be addressed in this fabrication technique: surface dimpling, and surface porosity.

TABLE 2-2. NO OVERLAY CONCEPT FABRICATION EVALUATION[a]

Sheet Material	0.152m x 0.152m (6" x 6") Weight kg x 10^3 (lb)	PBI Foam (64 kg/m^3 nominal) 0.152m x 0.152m x 0.0127m (6" x 6" x 1/2") Tile Weight, kg x 10^3 (lb)	Weight of Cured Panel Skinned 2 Sides, kg x 10^3 (lb)	Skin Weights Added, kg x 10^3 (lb)	PBI Weight, kg x 10^3 (lb)	Shear Test Results Test Area 0.0508m x 0.152m (2" x 6")
E-GLASS						
104 Fabric	0.46 (0.0010)	22.29 (0.0491)	28.07* (0.0619)	5.78 (0.0127)	4.86 (0.0107)	Core failure @ 356 kg (785 lb)
Surface Mat	0.69 (0.0015)	21.58 (0.0476)	26.31** (0.0580)	4.73 (0.0104)	3.35 (0.0074)	Core failure @ 374 kg (825 lb)
Surface Mat	0.69 (0.0015)	21.70 (0.0475)	26.91** (0.0593)	5.21 (0.0115)	3.83 (0.0084)	Core failure @ 313 kg (690 lb)

* Dimpled
** Smooth surfaces

[a] 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") PBI tiles with two layers of sheeting material

Surface porosity can be reduced by:

- Increasing the weight of PBI in the skin
- Using a denser mat

Efforts demonstrated that even after a several fold increase in the weight of PBI, the holes and porosity in industrial mat skinned tile were difficult to eliminate due to the high porosity of the mat. Subsequently, a denser mat was investigated. Unfortunately, mats or felts which would be suitable for use with the PBI system are not readily available. Commercially available Astroquartz mat, for example, is not sold in 2.54×10^{-4} to 5.08×10^{-4} m (10 to 20 mil) thicknesses. However, it can be pealed to 3.18×10^{-3} m ~(1/8 inch) thicknesses. Using this technique, the following result was obtained:

Weight of 0.152 m x 0.152 m x 0.0032 m (6" x 6" x 1/8") Astroquartz Mat, kg (lb)	Weight of Mat with PBI, kg (lb)
0.00079 (0.0017)	0.00496 (0.0109)

This weight exceeds the ~0.0025 kg (0.0055 lb) (one skin) suggested weight demonstrated previously (Table 2-2), however, when a smaller amount of PBI prepolymer was used with the mat, a coatable substrate was not obtained. It contained dry fibers. This small weight penalty may reflect a preferred skinning technique, however, for the same 0.0049 kg (0.0108 lb) obtained with the PBI/Astroquartz, two layers of a lightweight mat or fabric/mat combination can be used.

The double lightweight skin approach was therefore investigated and the following results obtained from preliminary experiments.

Weight (kg) of 0.152 m x 0.152 m (6" x 6") Square of Double Layer PBI Sheets	
1. Industrial Mat/Carbon felt	0.00374 kg (0.0082 lb)
2. Industrial Mat/104 E-glass fabric	0.00342 kg (0.0075 lb)

The 0.152 m x 0.152 m (6" x 6") square weights obtained from these two attempts in combination with the low porosity (visual) suggested that this was a good approach. It was concluded that this approach provided surface suitable for most types of coatings.

As a result of these fabrication evaluations, the Industrial Mat/E-glass fabric skin appeared to provide the lightest practical weight and minimized the porosity providing low permeability and a coatable dimple free surface.

However, the 104 E-glass was found difficult to work with and 108 E-glass with its better handleability was selected as the program baseline. The 104 E-glass [0.152 m x 0.152 m] (6" x 6") swatch weighs 0.00046 kg (0.0010 lb) and the 108 E-glass weighs 0.00108 kg (0.0024 lb).

As a result of the fabrication studies, the baseline tile composition, contrasted in Figure 2-2 with the reference tile concept, was selected. The manufacturing process associated with this tile concept is:

1. Impregnate surface mat with PBI adhesive
2. Wrap PBI foam billet with impregnated mat and cure in tile mold to 672°K (750°F) (Figure 2-3)
3. Impregnate 108 E-glass fabric
4. Place impregnated 108 E-glass fabric into bottom of mold and cure to 672°K (750°F)
5. Cured E-glass/mat/PBI tile at 728°K (850°F) for 1 hour.

2.3.2.1.5 Baseline Tile Fabrication Experience

Several tiles were fabricated to demonstrate the soundness of this approach and to provide a basis for weight comparisons with the predicted tile weights summarized in Table 2-3.

The details of fabrication of these tiles are given in Tables 2-4 and 2-5. Only three discrepancies have been noted for the cured tiles described in Table 2-4.

1. Slight warping of the tile
2. Residual porosity in the PBI/108 E-glass fabric top surface
3. Higher weights than predicted

The slight tile warping problem is due to the mismatch coefficients of expansion between the top surface (108 E-glass/mat) and bottom surface (mat) constructions. The amount of tile warpage depends upon both tile cross-sectional size and thickness. The warpage observed in the preliminary tile fabrications did not exceed ±0.020".

The residual porosity in the 108 E-glass top surface was minimal. Fabrication experience gained at this point demonstrated that the porosity did not affect the coatability of the tile as the various coatings could be applied with reproducable weights. This indicated that the coatings could not penetrate the residual porosity.

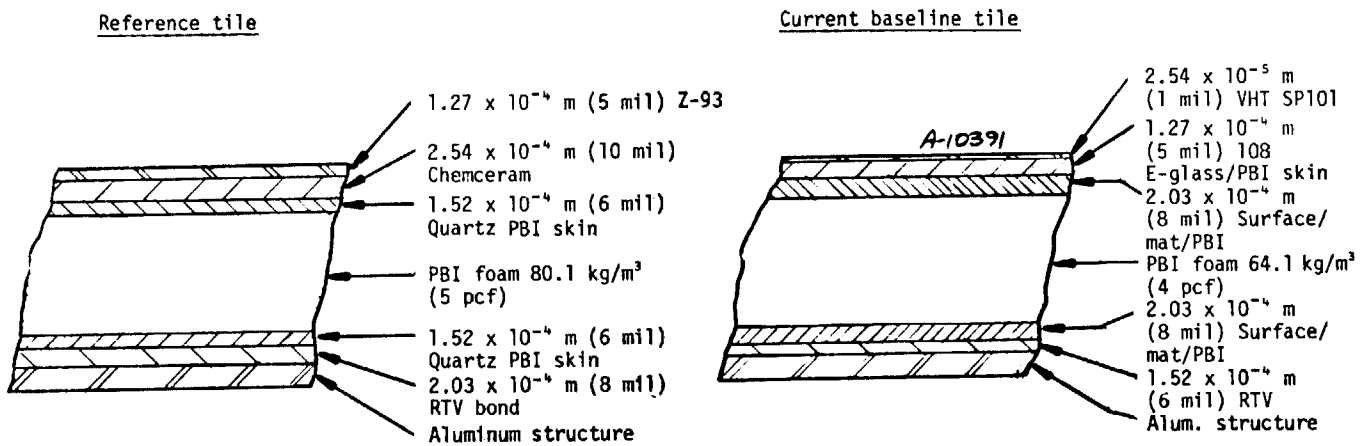


Figure 2-2. Comparison of reference and current PBI LRSI tile.

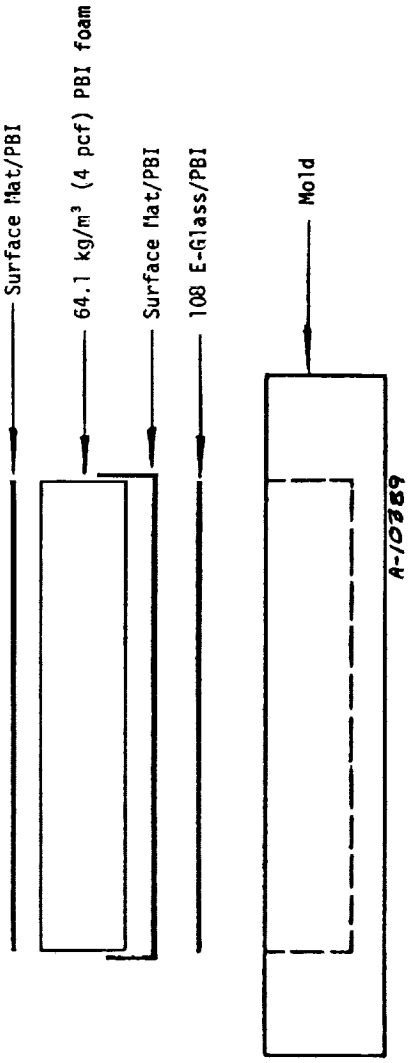


Figure 2-3. Layup technique for skinning tiles.

TABLE 2-3. PREDICTED BASELINE TILE WEIGHT*

PBI Foam Density kg/m^3 (lb/ft^3)	0.152m x 0.152m x 0.0127m (6" x 6" x 1/2") PBI Foam Tile Weight, kg x 10^3 (lb)	PBI/Mat Skins Weight kg x 10^3 (lb)	108 E-Glass/PBI Weight kg x 10^3 (lb)	Baseline PBI Tile Weight kg x 10^3 (lb)
56.1 (3.5)	16.55 (0.0365)	~5.0 (~0.011)	~3.0 (~0.0066)	24.55 (0.0561) [1.074 kg/m^2 (0.22 lb/ft^2)]
64.1 (4.0)	18.91 (0.0417)	~5.0 (~0.011)	~3.0 (~0.0066)	26.91 (0.0593) [1.172 kg/m^2 (0.24 lb/ft^2)]

*Prior to optical coating

TABLE 2-4. PBI PANEL WEIGHT COMPARISONS
(0.152 m x 0.152 m x 0.0127 m tile)
All Weights in kg x 10^3 (lbs)

Panel No.	Skin Composition		Foam Weight	Cured Panel Weight
	Top	Bottom		
1775-1 Panel 1	108/surface mat	Surface mat	19.4 (0.0428)	29.16 (0.0643)
1875-1 Panel 2		Surface mat	20.4 (0.0450)	31.35 (0.0689)
11375-1 Panel 3		108 and surface mat	--	32.8 (0.0723)
11475-1 Panel 4		Surface mat	19.85 (0.0438)	29.7 (0.0655)
11775-1 Panel 5			19.6 (0.0432)	33.4 (0.0736)
12075-1 Panel 6			22.15 (0.0488)	34.15 (0.0753)
12175-1 Panel 7			--	28.9 (0.0637)
12375-1 Panel 8			--	33.8 (0.0745)
12875-1 Panel 9			--	29.4 (0.0648)
12975-1 Panel 10			18.95 (0.0418)	31.9 (0.0703)
13175-1 Panel 11	Surface mat		21.32 (0.0470)	30.15 (0.0665)
12375-1 Panel 12	Surface mat Second cure 108			30.5 (0.0672) 32.4 (0.0714)

TABLE 2-5. PBI PANEL PROCESSING

Panel No.	Processing Conditions		Flatness	Comments
	Press Cure	Postcure N_2		
1775-1 Panel 1	1 hr 775°F	Oven 1 hr 900°F	Slight curvature	Dimples after postcure, VHT coated, cut in half and sent to NASA (0.0307)[3]
1875-1 Panel 2	1 hr 850°F	--	--	Surface had small dimples
11375-1 Panel 3	1 hr 750°F	--	No curvature	VHT paint cure 30 min @ 589K warped after VHT coating (0.0352)[3]
11475-1 Panel 4	1 hr 800°F	Oven 1 hr 850°F	--	Crack in 108 skin, panel flat (0.0303)[3]
11775-1 Panel 5	1 hr 750°F	Press 1 hr 925°F	Slight curvature	Skin dimpled. Weight after postcure 0.03206 kg
12075-1 Panel 6	1 hr 750	Oven 1 hr 850°F	No curvature	Surface smooth (0.0352)[3]
12175-1 Panel 7	1 hr 750°F	Oven 1 hr 850°F	Very slight curvature	Surface smooth 1.27×10^{-4}m (0.005")
12375-1 Panel 8	1 hr 750°F	Oven 1 hr 850°F	No curvature	Some wrinkles down sides 108, warped after several days
12875-1 Panel 9	1 hr 800°F	--	--	VHT coated cure 30 min @ 600°F. Sent to NASA (0.0307)[3]
12975-1 Panel 10	1 hr 850°F	--	--	108 cured skin wrinkled, was high in center, flat now (0.0344)[3]
13175-1 Panel 11	1 hr 850°F	--	Flat	Warped after VHT coating, some porosity in coating
12375-1 Panel 12	1 hr 850°F	--	--	Panel flat after surface mat cure (cure 1 hr @ 644K (700°K)). High in center after 108 cure (0.0359)[3]

NOTES: 1. 108 Fabric/PBI precured prior to lay-up.

2. Surface mat/foam cure precured prior to final lay-up.

3. Weight in kg after VHT coating

The weights of the fabricated tiles were slightly higher than the predicted tile weights ($\approx$ 4 g for a 0.152 m x 0.152 m x 0.0127 m tile). The calculated weights however are not representative of the fabrication technique. The PBI foam billet is actually cut oversize to insure that upon mold closure, the foam will snug the PBI surface mat against the mold surface. This accounts for approximately a 1 gram increase in foam weight. Control of the E-glass and mat fabric impregnation with the PBI prepolymer can further reduce the weight discrepancy.

With the baseline fabrication technique established, the manufacture of larger PBI tiles was investigated. Two 0.203m x 0.203m x 0.0127m (8" x 8" x 1/2") tiles were fabricated; one tile had a foam core density of 49.7 kg/m^3 (3.1 pcf) and the other a core density of 57.7 kg/m^3 (3.6 pcf). The low density tile exhibited surface dimpling whereas the 57.7 kg/m^3 (3.6 pcf) core specimen had an excellent surface character. Excess PBI present in the skins increased the tile weight to 0.051 kg (0.113 lb) from the expected weight of (extrapolated from previous 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") tile weights) 0.048 kg (0.106 lb). The amount of PBI powder deposited on the fabric, and the speed at which the fabric is pulled through the heated "doctor" blade are the manufacturing processes which must be controlled to assure reproducible skin impregnation. This had been accomplished in previous mat/skin efforts and does not present a problem for the fabrication of larger tiles.

Subsequent to the baseline PBI tile fabrication technique selection, fabrication of radiant lamp test specimens of various sizes has demonstrated the reproducability, dimensional tolerances, and tile sizes which result from the baseline tile fabrication technique. A total 16 test specimens were fabricated with 14 tiles having tolerances on all dimensions of 5.08 x 10^{-4} m ($\pm$0.020 in.). Table 2-6 summarizes the tile fabrication results and traces the processing results on a typical tile.

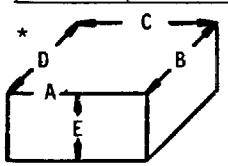
The tile fabrication efforts have demonstrated that the baseline no overlay tile concept provides an optimum weight system which has excellent surface characteristics for subsequent thermal control coating application. This fabrication technique has been demonstrated to be applicable to tiles of various sizes.

2.3.2.2 Candidate Thermal Control Coating Fabrication Evaluation

Samples of various candidate thermal control coatings were fabricated to evaluate the coating characteristics (e.g., weight, optical properties and mechanical properties). This section summarizes the results of these fabrication evaluations.

TABLE 2-6a. DETAILS OF PBI TILE FABRICATIONS (0.152 m x 0.152 m x 0.0127 m [6" x 6" x 1/2"])

Tile Number	Dry Weight			Environment Equilibrated Weight (kilograms)	Dry Tile Dimensions*		Coating Weight (kilograms)
	(kilograms)	(kg/m^2)	(lb/ft^2)		(meters)	(inches)	
12	30.3×10^{-3}	1.32	0.27	32.3×10^{-3}	0.152	6.00[a]	1.9×10^{-3}
					0.152	6.00[b]	
					0.152	6.00[c]	
					0.152	6.00[d]	
					0.0124	0.49[e]	
14	30.8×10^{-3}	1.32	0.27	32.9×10^{-3}	0.152	5.98[a]	1.8×10^{-3}
					0.152	5.98[b]	
					0.152	5.99[c]	
					0.152	6.00[d]	
					0.0124	0.49[e]	
15	30.2×10^{-3}	1.32	0.27	32.1×10^{-3}	0.152	5.99[a]	2.0×10^{-3}
					0.152	5.99[b]	
					0.152	5.99[c]	
					0.152	5.99[d]	
					0.0124	0.49[e]	
16	29.3×10^{-3}	1.27	0.26	31.4×10^{-4}	0.152	5.99 [a]	1.7×10^{-3}
					0.152	5.99[b]	
					0.152	5.99[c]	
					0.152	5.99[d]	
					0.0124	0.49[e]	



e.g., A
B
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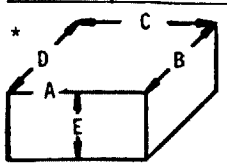
TABLE 2-6b. DETAILS OF PBI TILE FABRICATIONS (0.152 m x 0.152 m x 0.0254 m [6" x 6" x 1"])

Tile Number	Dry Weight			Environment Equilibrated Weight (kilograms)	Dry Tile Dimensions*		Coating Weight (kilograms)
	(kilograms)	(kg/m^2)	(lb/ft^2)		(meters)	(inches)	
6	48.1×10^{-3}	2.05	0.42	51.5×10^{-3}	0.152	5.99[a]	2.0×10^{-3}
					0.152	5.99[b]	
					0.152	6.00[c]	
					0.152	6.00[d]	
					0.0251	0.99[e]	
7	5.13×10^{-3}	2.20	0.45	54.9×10^{-3}	0.152	5.99[a]	2.0×10^{-3}
					0.152	5.99[b]	
					0.152	5.99[c]	
					0.152	5.99[d]	
					0.0251	0.99[e]	
9	48.7×10^{-3}	2.10	0.43	52.3×10^{-3}	0.152	5.98[a]	1.7×10^{-3}
					0.152	5.98[b]	
					0.153	5.98[c]	
					0.152	5.98[d]	
					0.0251	0.99[e]	
19	50.1×10^{-3}	2.15	0.44	52.6×10^{-3}	0.152	5.98[a]	2.5×10^{-3}
					0.152	5.98[b]	
					0.152	5.98[c]	
					0.152	5.98[d]	
					0.0251	0.99[e]	
4	47.6×10^{-3}	2.05	0.42	50.9×10^{-3}	0.151	5.94[a]	1.8×10^{-3}
					0.152	5.97[b]	
					0.152	5.97[c]	
					0.152	5.97[d]	
					0.0251	0.99[e]	

* 

e.g., A
B
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TABLE 2-6c. DETAILS OF PBI TILE FABRICATIONS (0.203 m x 0.203 m x 0.0127 m [8" x 8" x 1/2"])

Tile Number	Dry Weight			Environment Equilibrated Weight (kilograms)	Dry Tile Dimensions*		Coating Weight (kilograms)
	(kilograms)	(kg/m^2)	(lb/ft^2)		(meters)	(inches)	
8	52.6×10^{-3}	1.27	0.26	56.4×10^{-3}	0.203	8.00[a]	2.5×10^{-3}
					0.203	8.00[b]	
					0.203	8.00[c]	
					0.203	8.00[d]	
					0.0124	0.49[e]	
11	53.0×10^{-3}	1.27	0.26	56.8×10^{-3}	0.203	7.98[a]	2.7×10^{-3}
					0.203	8.00[b]	
					0.203	8.00[c]	
					0.203	8.00[d]	
					0.0124	0.49[e]	
17	53.3×10^{-3}	1.27	0.26	57.1×10^{-3}	0.203	8.00[a]	2.6×10^{-3}
					0.203	8.00[b]	
					0.203	8.00[c]	
					0.203	8.00[d]	
					0.0124	0.49[e]	
18	50.3×10^{-3}	1.22	0.25	53.7×10^{-3}	0.203	7.99[a]	3.9×10^{-3}
					0.203	7.99[b]	
					0.203	7.99[c]	
					0.203	8.00[d]	
					0.0124	0.49[e]	
10	51.1×10^{-3}	1.22	0.25	54.6×10^{-3}	0.203	7.99[a]	3.7×10^{-3}
					0.203	7.99[b]	
					0.203	7.99[c]	
					0.203	7.99[d]	
					0.0124	0.49[e]	
13	51.3×10^{-3}	1.22	0.25	54.0×10^{-3}	0.203	8.00[a]	2.9×10^{-3}
					0.203	8.01[b]	
					0.203	8.01[c]	
					0.203	8.01[d]	
					0.0124	0.49[e]	

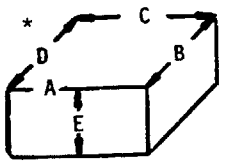

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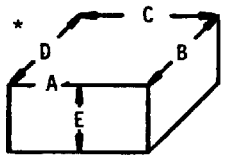
TABLE 2-6d. DETAILS OF PBI TILE FABRICATIONS (0.203 m x 0.203 m x 0.0254 m [8" x 8" x 1"])

Tile Number	Dry Weight			Environment Equilibrated Weight (kilograms)	Dry Tile Dimensions*		Coating Weight (kilograms)
	(kilograms)	(kg/m^2)	(lb/ft^2)		(meters)	(inches)	
1	85.5×10^{-3}	2.05	0.42	91.4×10^{-3}	0.203	7.99[a]	3.4×10^{-3}
					0.203	7.99[b]	
					0.23	7.99[c]	
					0.23	7.99[d]	
					0.0251	0.99[e]	
5	88.3×10^{-3}	2.15	0.44	94.3×10^{-3}	0.203	7.99[a]	3.0×10^{-3}
					0.203	7.99[b]	
					0.203	7.99[c]	
					0.203	7.99[d]	
					0.0251	0.99[e]	

* 

e.g., A
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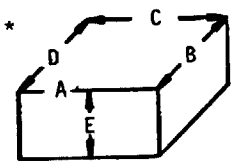
TABLE 2-6e. DETAILS OF PBI TILE FABRICATIONS (0.305 m x 0.305 m x 0.0254 m [12" x 12" x 1"])

Tile Number	Dry Weight			Environment Equilibrated Weight (kilograms)	Dry Tile Dimensions*		Coating Weight (kilograms)
	(kilograms)	(kg/m^2)	(lb/ft^2)		(meters)	(inches)	
2	195.1×10^{-3}	2.10	0.43	209.1×10^{-3}	0.303	11.93[a]	7.6×10^{-3}
					0.303	11.93[b]	
					0.303	11.94[c]	
					0.303	11.94[d]	
					0.0251	0.99[e]	
3	197.0×10^{-3}	2.10	0.43	210.9×10^{-3}	0.304	11.97[a]	5.7×10^{-3}
					0.304	11.97[b]	
					0.304	11.98[c]	
					0.305	12.00[d]	
					0.0251	0.99[e]	

* 

e.g., A B C D E

TABLE 2-6f. FABRICATION DETAILS OF A TYPICAL TILE (0.152m x 0.152m x 0.0127m [6" x 6" x 1/2"])

Foam Weight After 2 hr @ 422K		Surface Mat/PBI Weight	108E Glass/PBI Weight	VHT SP101 Coating Weight	Finished Tile Weight
Weight kg	Dimensions m	kg	kg	kg	kg
2.07×10^{-2}	0.153 m	5.2×10^{-3}	2.5×10^{-3}	1.9×10^{-3}	30.3×10^{-3}
	0.153 m				
	0.153 m				
	0.153 m				
	0.0132 m				

The thermal control coating samples described below generally consisted of a layer of surface mat and a layer of 108 E-glass impregnated with PBI and cured together through a minimum of 672°K (750°F). The PBI/108 E-glass surface was then sanded and the candidate thermal control coating applied.

VHT SP101 White

Two VHT SP101 922°K (1200°F) coated skins were fabricated with the following characteristics (for 0.152 m x 0.152 m (6" x 6") skin).

Mat/108 Skin Weight, kg (lbs)	Cured Weight, kg (lbs)	VHT Weight, kg (lbs)
0.00546 (0.0120)	0.00782 (0.0172)	0.00236 (0.0052)
0.00542 (0.0119)	0.00697 (0.0154)	0.00155 (0.0034)

The VHT SP101 is sold by Sperex Corporation of Gardena, California and consists of a GE silicone which is modified by Sperex to provide a low solar absorption coefficient and remain stable at 922°K (1200°F) in air.

VHT SP101 on Glass Fabrics

Samples of glass were coated with VHT SP101 for optical property testing. The coatings were applied with two techniques: spray coated and dip coated. The spray coating was applied with commercially available spray cans held at a distance of between 0.305 m and 0.457 m (12 to 18 inches) from the surface. For the dip coating, swatches of fabric were dipped into a container of VHT SP101 two or three times until visual coverage was obtained. The details of the fabrication results are reported below.

Fabric Style	Fabric Weight kg x 10^3 (lbs x 10^{-3})	VHT/Fabric Weight kg x 10^3 (lbs x 10^{-3})
	Spray Coated	
108	0.44 (0.00097)	1.86 (0.0041)
181	3.35 (0.00739)	4.44 (0.0098)
	Dip Coated	
104	0.16 (0.00035)	0.30 (0.00066)
104*	0.073 (0.00016)	0.19 (0.00042)
108	0.58 (0.00128)	0.78 (0.00172)
108*	0.20 (0.00044)	0.34 (0.00075)
181	3.35 (0.00738)	4.47 (0.0098)
181*	1.16 (0.00256)	1.83 (0.0040)

*0.0254 m x 0.152 m (1" x 6")

Flame Sprayed Coatings

Four 0.152 m x 0.152 m (6" x 6") sheets of 108 E-Glass/Mat PBI skins were forwarded to Ametek-Straze for flame spraying and if successful, two weights of the zirconia and alumina coatings were to be applied. Standard flame spray techniques were employed varying the airflow, rate of rod erosion (alumina), distance of torch from substrate, and substrate preparation to obtain a uniformly coated surface. The details are as listed below.

Panel Number	Wt. of PBI Skin, kg x 10^3 (lbs)	Wt. of Aluminum Oxide, kg x 10^3 (lbs)	Thickness, m (mils)	
1	6.7 (0.0148)	21.0 (0.0463)	5.59×10^{-4} - 6.35×10^{-4}	(22-25)
2	5.87 (0.0129)	20.8 (0.0459)	4.83×10^{-4} - 5.08×10^{-4}	(19-20)
3	5.0 (0.001)	20.3 (0.0448)	4.32×10^{-4} - 5.08×10^{-4}	(17-20)
4	5.5 (0.0121)	26.3 (0.0580)	5.33×10^{-4} - 6.86×10^{-4}	(21-27)

Flame sprayed zirconia abraided the PBI skins during application and consequently is an unsuitable coating.

Sauereisen and Sauereisen/Fabric Coatings

A mixture of 19.6 percent Sauereisen No. 29, 70 percent Sauereisen No. 14 Thinner and 10.4 percent Emerson & Cumings FTF.15 glass bubbles was coated onto two 0.152 m x 0.152 m (6" x 6") sheets of 108/Mat/PBI at two weights. The Sauereisen coatings were troweled onto the sheets in accordance with the manufacturer's specifications to a thickness which provided visual coverage. The dried weights of the two coatings were 0.012 and 0.018 kg (0.0265 and 0.0397 lbs). These coatings debonded upon drying at 373°K (211°F).

The Sauereisen Company was contacted for assistance in techniques which would provide better adhesion to the PBI. Sauereisen cements do not adhere well to organics, but the use of a silane coupling agent sometimes improves the adhesion. The Sauereisens can be pigmented and they do require pigmenting in order to reduce the coating weight required to whiten the PBI. Three Sauereisen coated glass fabrics were fabricated with the following results:

Fabric	Sample Weight 0.152 m x 0.152 m (6" x 6") kg (lb)
108 E-glass	0.0126 (0.0278)
116 E-glass	0.0134 (0.0295)
181 E-glass	0.0180 (0.0397)

Optical property measurements were made on these samples. However, the coatings were extremely brittle and are unsuitable for use on the baseline tile even if better adhesion to the PBI can be accomplished with a coupling agent.

DC 805 Silicone

DC 805 silicone resin and TiO_2 (-325 mesh) coatings have been applied successfully to the prepared PBI skins. A solution of toluene, DC 805 resin and TiO_2 pigment was sprayed onto the sanded substrate from a distance of approximately 0.305 m (12 inches). The DC 805 was filled with TiO_2 at two weight levels: one part of resin to two and three parts of TiO_2. The data for the two 0.152 m x 0.152 m (6" x 6") skins are as follows:

	Weights, kg (lbs)	
TiO_2 Loading	1 to 2	1 to 3
Wt. of PBI skins	0.0066 (0.0146)	0.0064 (0.0141)
Coating	0.0050 (0.0110)	0.0036 (0.0070)
Total	0.0116 (0.0256)	0.0100 (0.0220)

Cured: 40 minutes at 533°K (500°F)

From a fabrication standpoint, this coating system is acceptable.

A review of the coating fabrication work reveals that three acceptable coating concepts can be identified. These are (listed in order of increasing weight):

1. VHT (SP101)
2. DC 805
3. Flame sprayed alumina

2.3.3 Foam Moisture Evaluation

PBI foam has a residual moisture content of approximately 4 percent under normal atmospheric conditions. This water is removed under vacuum exposure or by heating. Treatment of the foam with known organic hydrophobic compounds was investigated in an attempt to reduce this moisture content. It is desirable to reduce the equilibrium moisture content of the tiles for several reasons. First, lower moisture content tiles are lighter, and secondly, when subjected to ascent heating and depressurization, internal pressures caused by moisture vaporization are reduced. Thirdly, tile warpage is reduced when the foam moisture content is limited. PBI samples were skinned on two surfaces leaving the sides open for treatment with the hydrophobes. The hydrophobes were dissolved in a low boiling temperature solvent, and the test sample was submerged in the solution until air stopped coming out of the specimen. The sample was subsequently dried. The residual moisture content of each sample was established prior to hydrophobic treatment. Subsequent to the treatment, the samples were allowed to equilibrate with a controlled humidity environment, and the resultant weight

gain from moisture pickup was obtained. In addition, several specimens were heated to 617°K (650°F) for 1 hour. The equilibrium moisture contents were again established. These test results are presented in Table 2-7. Treatment of the foam with the hydrophobic agents did not significantly affect the equilibrium moisture content, and the total weight of the material was in general increased by the amount of hydrophobic compound added.

The five silicone treated samples seen in Table 2-7 which had been heated to 617°K (650°F) were tested for resistance to penetration by liquid water. Two drops of water were placed on a surface and allowed to stand for 15 seconds. The drops were blown off with N_2 and the tile was reweighed. These results are presented in Table 2-7a. It is concluded that the DC 530 provides a waterproofing effect suggesting good rain resistance with a tile weight penalty due to the treatment of from 5.4 to 7.5 percent.

Additional testing has been performed to evaluate the moisture resistance of the baseline tile concept in both the coated and uncoated configurations. Four 0.152 m x 0.152 m x 0.0127 x (6" x 6" x 1/2") tiles were weighed, dried, and reweighed to obtain the equilibrium moisture content of the PBI tiles. In addition, weight loss of the tiles between their previous cure history 672°K (750°F) and an additional 728°K (850°F) cure was recorded. These results are presented in Table 2-8. The four tiles showed an average moisture loss of 6.1 percent after 1 hour at 450°K (350°F). The tiles had an average weight loss, after the 728°K (850°F) cure, of 0.3 percent with two showing essentially no loss which indicates the mass loss in the cure cycle is basically completed at a temperature of 672°K (750°F). The average moisture regains of the four tiles was 5.3 percent.

The 5.3 percent is a nominal 1 percent higher than has been measured on the PBI foam in nontile geometry. The small amount of glass added to tile may be responsible for this 1 percent increase. It requires about 2 days for the tiles to attain their equilibrium weights.

Tile numbers 4 and 6 were subsequently coated with the VHT SP101. Tile No. 4 was coated with 1.0 g and No. 6 with 1.7 g of the coating. The equilibrium weight increases of the tiles after the coating had been cured were 4.6 percent and 4.2 percent, respectively, showing 0.5 percent to 1.6 percent weight savings over the uncoated tiles.

TABLE 2-7. MOISTURE PICKUP OF SKINNED PBI TILES
(Equilibrium weights; weights in kg x 10^3 (lb))

Treatment/% Wt. Increase Due to Additive	Dry Sample Weight	Weight Increase of Control		Weight Increase After Treatment
		kg x 10^3 (lb)	%	
Nujol/18.5	0.8573 (1.89 x 10^{-3})	0.0325 (7.16 x 10^{-5})	3.79	0.0335 (7.39 x 10^{-5})
Silicone Oil/43.1 (DC 710) After 617°K (650°F)/38.0	0.7317 (1.61 x 10^{-3})	0.0295 (6.50 x 10^{-5})	4.0	0.0289 (6.37 x 10^{-5}) 0.0381 (8.40 x 10^{-5})
Sulfonated Aliphatic Hydrocarbon/17.7	0.8709 (1.92 x 10^{-3})	0.0297 (6.55 x 10^{-5})	3.40	0.0561 (1.24 x 10^{-4})
Stearic Acid/13.6	0.7906 (1.74 x 10^{-3})	0.0336 (7.41 x 10^{-5})	4.24	0.0315 (6.94 x 10^{-5})
Silicone DC473/124.1 After 617°K (650°F)/71.2	0.7989 (1.70 x 10^{-3})	0.0253 (5.58 x 10^{-5})	3.28	0.0374 (8.24 x 10^{-5}) 0.0465 (1.02 x 10^{-4})
Silicone F754/64.6 After 617°K (650°F)/36.3	0.8442 (1.86 x 10^{-3})	0.0345 (7.61 x 10^{-5})	4.08	0.0498 (1.10 x 10^{-4}) 0.0559 (1.23 x 10^{-4})
Silicone F755/81.6 After 617°K (650°F)/54.3	0.8112 (1.79 x 10^{-3})	0.0375 (8.27 x 10^{-5})	4.02	0.0324 (7.14 x 10^{-5}) 0.0428 (9.44 x 10^{-5})
Silicone DC530/7.1 After 617°K (650°F)/5.4	0.8608 (1.90 x 10^{-3})	0.0327 (7.21 x 10^{-5})	3.79	0.0370 (8.16 x 10^{-5}) 0.0469 (1.03 x 10^{-4})

TABLE 2-7a. LIQUID MOISTURE RESISTANCE OF SKINNED PBI TILES
(Weight in kg x 10^3 (lb))

Treatment	Original Sample Weight	Sample Weight After Treatment	Change in Sample Weight	
Standard	0.8950 (0.00197) 0.7541 (0.00166)	0.8974 (0.00198) 0.7561 (0.00167)	0.0024 (5.29×10^{-6}) 0.0020 (4.40×10^{-6})	Slight wetting
F-755	1.2963 (0.00286) 1.2121 (0.00267)	1.2979 (0.00286) 1.2123 (0.00267)	0.0016 (3.52×10^{-6}) 0.0002 (4.40×10^{-7})	Very poor wetting
DC-473	1.3586 (0.00300) 1.3923 (0.00307)	1.3821 (0.00305) 1.3979 (0.00308)	0.0235 (5.18×10^{-5}) 0.0056 (1.23×10^{-5})	Good wetting
DC-530	0.7813 (0.00172) 0.9594 (0.00212)	0.7814 (0.00172) 0.9595 (0.00212)	0.0001 (2.2×10^{-7}) 0.0001 (2.2×10^{-7})	Very poor wetting
F-754	0.9094 (0.00200) 1.2024 (0.00265)	0.9446 (0.00208) 1.2328 (0.00272)	0.0352 (7.75×10^{-5}) 0.0304 (6.7×10^{-5})	Good wetting
DC-710	1.0561 (0.00233) 1.1141 (0.00246)	1.0583 (0.00233) 1.1211 (0.00247)	0.0022 (4.84×10^{-6}) 0.0070 (1.54×10^{-5})	Unreproducible wetting — some wet, some don't

TABLE 2-8. PBI TILE WEIGHT LOSS AND MOISTURE REGAIN AFTER 728°K (850°F) CURE*

Measurement	Tile Number 4	Tile Number 6	Tile Number 7	Tile Number 8
Starting tile weight	31.25 (0.0689)	35.68 (0.0787)	30.45 (0.0671)	35.70 (0.0787)
Weight after oven dry [% loss]	29.43 (0.0649) [6.2]	33.62 (0.0771) [6.2]	28.9 (0.0637) [5.3]	33.45 (0.0737) [6.7]
Weight after 1 hour @ 728°K (850°F)[% loss]	29.35 (0.0647) [0]	33.50 (0.0738) [0.4]	28.70 (0.0633) [0.7]	33.47 (0.0738) [0]
Weight after ambient aging [% regain]:				
2 days	30.85 (0.0680)	35.24 (0.0777)	–	–
3 days	30.85 (0.0680) [5.1]	35.45 (0.0782) [5.8]	–	–
4 days	–	–	30.2 (0.0666)	35.3 (0.0778)
5 days	–	–	30.1 (0.0664) [4.9]	35.2 (0.0776) [5.2]

*0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2")

2.3.4 Screening Tests

Throughout the coating development program, screening tests have been performed to evaluate the applicability of various thermal control coating and foam packaging concepts. The foam packaging concepts must provide foam stabilization, survive thermal shock, have structural integrity, have adequate impact resistance, and provide an appropriate substrate for the thermal control coating. In addition, the thermal control coating must provide the correct optical properties, demonstrate water proofing capability, and survive the ground handling environment.

A variety of screening tests have been performed to evaluate the candidate packaging and thermal control coating comcepts. Section 2.3.4.1 presents the results of shear testing and Section 2.3.4.2 reviews the thermal shock testing. The impact testing is evaluated in Section 2.3.4.3. Outgassing test results are presented in Section 2.3.4.4 and optical property measurements of the candidate thermal control coatings are reviewed in Section 2.3.4.5.

2.3.4.1 Shear Testing

Shear tests were used to evaluate the bond between the foam and its skins. The shear characteristics of the candidate skin/foam package concepts were evaluated in tests similar to those decribed in ASTM C273. The preliminary screening was performed at room temperature with the panels bonded to the loading plates.

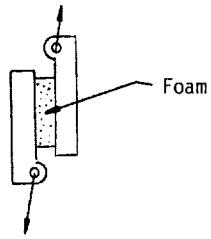


Table 2-9 presents a summary of these results for both integral and bonded skin tile construction. The test results all indicate acceptable skin fabrication techniques as core failure was observed without skin delamination from the foam. Additional shear testing has been performed on the selected tile packaging technique at elevated temperatures to assess the skin/foam bond integrity under conditions typical of a shuttle entry. Table 2-10 presents the results of these tests. These results demonstrate that the foam is the weakest part of the baseline tile at temperature.

TABLE 2-9. PBI TILE SHEAR TEST RESULTS

Sheet Material	Skin Attachment Technique	Shear Test Results Test Area 0.0508m x 0.1524m (2" x 6")
104 E-Glass	PBI Adhesive	Core failure @ 293 kg (645 lb)
112 E-Glass	PBI Adhesive	Core failure @ 311 kg (685 lb)
116 E-Glass	PBI Adhesive	Core failure @ 395 kg (870 lb)
Surface Mat	PBI Adhesive	Core failure @ 333 kg (735 lb)
Swirled Mat	PBI Adhesive	Core failure @ 356 kg (785 lb)
Astro Mat Quartz	PBI Adhesive	Core failure @ 260 kg (575 lb)
104 E-Glass	Integral Bond	Core failure @ 356 kg (785 lb)
Surface Mat	Integral Bond	Core failure @ 374 kg (825 lb)
Surface Mat	Integral Bond	Core failure @ 313 kg (690 lb)

TABLE 2-10. TILE SHEAR TESTS

Test Temperature	Shear Test Results, 0.0508 m x 0.152 m (2" x 6") Test Area
RT	Core failure at 207 kg (457 lbs)
533°K (500°F)	Core failure at 194 kg (429 lbs)
644°K (700°F)	Adhesive failed at 67 kg (147 lbs)

2.3.4.2 Outgassing Tests

In order to verify the thermal stability of the PBI tile, outgassing tests were conducted on two elements of the tile by Dr. John Park at Goddard Space Center. These results were:

769°K (925°F) Cured PBI Foam

Average total mass loss	4.09 percent
Average condensables	0.01 percent

728°K (850°F) Cured Mat/Fabric/PBI skin

Average total mass loss	2.77 percent
Average condensables	0.02 percent

Regain After Conditioning

24 hours at RT in 50 percent R.H.	2.77 percent

These results demonstrate that the mass loss is primarily attributable to the loss of moisture from the tile components. In addition, less than 0.02 percent condensables were observed, which indicates that contamination of components surrouding a PBI tile will be virtually nonexistant for the reuse thermal envelope of interest ($T \leq 850°F$).

2.3.4.3 Optical Property Testing

Measurements of coating optical properties were required to characterize the response of the PBI system to the orbital and reentry thermal environments. For each coating material to be evaluated, tests were needed to evaluate the following properties:

Emittance versus surface temperatures for the range 294°K to 811°K (70°F to 1000°F)

Emittance following exposure at high temperature in an oxidizing environment

Solar absorptance

Solar absorptance following exposure to ultraviolet degradation

These optical measurements were performed by NASA Ames on the candidate coating concepts described in Section 2.3.2.2, and Table 2-11 presents a summary of these results. Flame sprayed alumina, and 181 glass overlay provide unacceptable optical properties throughout the temperature range of interest. In addition, after a thermal exposure of 16 hours at 617°K (650°F) the DC 805 with 3 parts TiO_2 and the VHT SP101 demonstrated acceptable optical performance. The thermal stability of the optical coating is a desirable feature (required for shuttle application). A review of the relative coating weights indicates that VHT SP101 is by far the lightest. An additional test has been performed to evaluate the flexibility of the various candidate thermal control coatings before and after thermal exposure. These results, presented in Table 2-12, indicate that VHT SP101 is the most flexible coating.

Evaluation of the optical coating application techniques, optical characteristics, and thermal stability resulted in the selection of a baseline optical coating system. The following discussion delineates the reasons for the elimination of various coating concepts. Flame sprayed zirconia abraided the PBI skins during application, and consequently was an unsuitable coating. The Sauereisen coatings did not bond well to the PBI skins, and hence were not appropeiate candidate coatings. The flame spray alumina coating was extremely heavy and difficult to fabricate and consequently was not an acceptable candidate. The VHT SP101 and the TiO_2 loaded DC 805 (2 parts TiO_2) both have acceptable optical properties, however, thermal stability testing indicated that DC 805 was significantly more brittle after heat soak at 617°K (650°F). Hence, VHT SP101 was selected as the prime candidate coating system.

2.3.4.4 Thermal Shock Testing

The thermal shock testing and impact testing evaluated the baseline system performance only (i.e., VHT SP101 was the only thermal control coating evaluated). The effects of low temperatures (typical of on-orbit environments) upon the tile integrity were investigated under this program using thermal shock testing on the baseline tile configuration coated with VHT SP101. In addition, the tiles were bonded to an aluminum plate and subjected to severe cold/thermal shock environments to assess the capabilities of the tile/bond system.

The sample was immersed in LN_2 for 15 minutes, removed and placed in a 644°K (700°F) oven. The tile was then allowed to cool to room temperature. After 10 such cycles, the surface exhibited microcracking in the VHT coating. Additional testing has indicated that the microcracking typically occurs in the high temperature portion of the thermal cycle (RT to 644°K (700°F)). Four thermal shock cycles of the baseline tile concept bonded with RTV-560 to a 1.78 kg (0.062 inch) aluminum plate were performed. The thermal shock cycles provided:

TABLE 2-11. OPTICAL COATING SELECTION**

Coating Concept	Optical Properties				Coating Weight kg/m^2 (lb/ft^2)	Comments
	α	ϵ				
		RT	700°K (800°F)	922°K (1200°F)		
Z-93	0.20	0.8			0.430 (0.088)	Reference
Sauereisen	0.25	0.9	-	0.88	0.518 (0.106)	Crazed, poor bond
Flame spray alumina	0.27	0.77	0.68	0.59	0.875 - 1.134 (0.179 - 0.232)	
Flame spray zirconia	-		-	-	-	PBI abraded
181 glass overlay	0.35		0.77	0.68	0.293 (0.060)	
DC805						
— 2 parts TiO_2	0.18 (0.25)*	0.87	0.87 (0.87)	0.84 (0.84)	0.215 (0.044)	Coated cracked
— 3 parts TiO_2	0.15 (0.18)	0.87	0.73 (0.73)	0.65 (0.65)	0.156 (0.032)	
VHT SP101	0.36	0.87	0.87 (0.87)	0.86 (0.86)	0.068 - 0.103 (0.014 - 0.021)	o Adheres well

*( ) indicates results after 16 hours at 617°K (650°F)

**tests performed by NASA/ARC

TABLE 2-12. PBI SKIN/OPTICAL COATING THERMAL STABILITY*

Optical Coating	Test Conditions		Radius to Break Meters (inches)
	Hrs	Temp K(°F)	
VHT-A		Pretest	0.0064 (0.25)
VHT-A	16	617 (650)	0.0064 (0.25)
VHT-B		Pretest	0.0048 (0.19)
VHT-B	16	617 (650)	0.0030 (0.12)
DC805 - 2 parts TiO_2		Pretest	0.0079 (0.31)
DC805 - 2 parts TiO_2	16	617 (650)	0.0254 (1.00)**
DC805 - 3 parts TiO_2		Pretest	0.0096 (0.38)
DC805 - 3 parts TiO_2	16	617 (650)	0.0127 (0.05)

*Tests performed by NASA/ARC

**Coating cracked

- 10 min. hold in LN_2 (72°K (-320°F))
- 2 min. hold in RT (294°K (70°F))
- 10 min. hold on 644°K (700°F) platten

These tests demonstrated the integrity of the baseline tile concept (including bond) in severe thermal shock environments.

2.3.4.5 Impact Testing

Preliminary screening tests have been performed to evaluate the impact resistance of the baseline tile system. These tests were performed by dropping a steel ball (0.0413 m (1-5/8 inch) diameter, 0.281 kg (0.62 lb)) onto a VHT SP101 coated tile from varying heights. The results of these tests are presented in Table 2-13.

TABLE 2-13. IMPACT RESISTANCE OF PBI TILE

Joules	Energy (ft-lb)	Effect
0.013	(0.31)	Skin torn
0.013	(0.31)	Skin torn
0.0084	(0.20)	Tile dented – recovered
0.0084	(0.20)	Tile dented – skin torn
0.0084	(0.20)	Tile dented – did not recover
0.0084	(0.20)	Tile dented – did not recover
0.0084	(0.20)	No visible effect
0.0067	(0.16)	No visible effect
0.0067	(0.16)	No visible effect

2.3.4.6 Adhesive Evaluation

Three adhesives, RTV-560, Hysol modified 934 and DC XF-3-6016 were tested for use as an adhesive for bonding PBI tiles to aluminum. After cure at room temperature the specimens were soaked for 15 minutes in LN_2 and then warmed to 450°K (350°F) in an oven. The RTV-560 and Hysol modified 934 were sawed into smaller specimens. The foam was pryed away from the aluminum, and the following was observed:

	Adhesion to Aluminum	Adhesive	Adhesion to Foam
RTV-560	No failure	Failure	No failure
Modified Hysol 934	Failure	No failure	No failure
DC XF-3-6016	Failure	No failure	No failure

Thus, the RTV-560 failed cohesively and the other two adhesively.

These results are interpreted as follows: The RTV-560 bonds well to both the aluminum and PBI foam; the PBI foam at this density ~96 kg/m^3 (6 pcf) is stronger than the RTV-560. The ideal adhesive would have caused the PBI to fail. Consequently, this test indicates that the RTV-560 may limit tile size since it fails prior to the PBI foam. These tests also indicate that the other adhesives are inferior to RTV-560 for bonding to the aluminum substrate. Thus, RTV-560 was the selected adhesive for the PBI system.

2.4 TASK 3 — MATERIAL PROPERTIES

The various components of the PBI system had to be characterized in order to provide the required data base for analyzing the performance of the coated PBI tile in projected operational environments (e.g., shuttle upper surface, hypersonic aircraft, space tug, etc.). Both mechanical and thermophysical data were obtained on each tile component through the following tests:

- Tensile strength
- Compression
- Thermal expansion
- Thermal conductivity

Section 2.4.1 reviews the results of the mechanical property testing, and thermophysical property evaluations are summarized in Section 2.4.2.

2.4.1 Mechanical Properties

Both tension and compression testing were performed on PBI foam over the temperature range wherein the PBI reuse capability exists. In addition to foam characterization, the mechanical properties of the baseline skin concepts were obtained from tensile tests performed over the same temperature range. Table 2-14 presents the results of the foam tensile testing (performed per ASTM D1623), and Table 2-15 reviews the compression test results (performed as per ASTM D1621). The matrix displayed in Table 2-15 indicates the results of testing performed on each individual specimen sectioned from a 0.3048 m (1 foot) by 0.3048 m (1 foot) PBI foam panel. Density, strength, direction of test, and modulus' are reported for each specimen tested.

The tensile test results for the two tile skin fabrications are summarized in Tables 2-16 and 2-17. These results indicate that the surface mat/PBI skin required approximately a factor of three lower load for failure than the 108 E-glass/surface mat/PBI skin construction.

TABLE 2-14. TENSILE STRENGTH OF PBI FOAM**

Thermal Exposure* Temperature, °K (°F)	Strength N/m^2 (psi)	Modulus N/m^2 (psi)	Strain to Failure m/m
RT	5.24×10^5 (76)	2.21×10^7 (3.2×10^3)	0.038
561 (550)	6.41×10^5 (93)	2.00×10^7 (2.9×10^3)	0.042
617 (650)	2.90×10^5 (42)	6.21×10^6 (0.9×10^3)	0.043
644 (700)	2.76×10^5 (40)	2.00×10^7 (2.9×10^3)	0.022

NOTES: 1. All properties averaged

2. PBI foam nominal density, 64 kg/m^3 (4 lbs/ft^3)

*16 hours at 5066 n/m^2 (0.05 atm) air pressure

**Tests performed by NASA/ARC

TABLE 2-15. COMPRESSION STRENGTH OF PBI FOAM*

North Side

42.46 30.61 ↕ 8.27	38.75 38.61 ⊙ 8.96	39.33 35.23 ↔ 10.34	40.14 26.68 ↕ 6.89	41.30 40.47 ○ 11.72	43.85 49.44 ↔ 15.86	40.03 30.61 ↕ 8.27	54.76 58.81 ○ 12.41
35.27 25.71 ⊙ 6.75	45.36 47.78 ↕ 14.48	44.78 44.75 ↔ 12.41	40.26 30.34 ○ 7.58	42.11 44.75 ↕ 14.48	45.13 45.37 ↔ 9.65	46.06 45.37 ⊙ 12.41	47.68 36.75 ↔ 9.65
37.82 26.68 ↔ 6.89	41.53 35.51 ↔ 10.34	38.75 30.89 ⊙ 6.75	42.00 41.37 ↔ 11.72	42.35 41.92 ↕ 11.72	42.35 33.37 ○ 8.27	44.90 46.54 ↕ 11.72	49.54 44.12 ○ 11.72
39.21 24.72 ↕ 8.96	36.89 28.82 ↕ 8.27	38.87 33.09 ↔ 8.96	40.26 32.13 ⊙ 7.58	43.97 42.89 ↕ 12.41	44.43 46.54 ↕ 13.79	47.22 50.26 ↔ 13.79	44.09 57.02 ↕ 19.30
36.78 22.06 ○ 6.34	37.47 26.96 ○ 6.55	37.00 30.61 ↕ 8.96	26.96 ○	42.86 ↔	46.41 49.64 ↔ 12.41	46.99 36.13 ○ 9.65	41.30 40.47 ↔ 13.10
40.37 32.47 ↔ 9.65	38.52 31.23 ↔ 8.96	38.98 25.10 ⊙ 6.89	41.65 37.37 ↔ 11.03	43.51 48.40 ↕ 15.17	42.93 34.89 ○ 8.96	42.58 47.16 ↕ 14.48	38.75 32.75 ◜ 8.27
38.40 29.72 ↕ 17.93	35.27 30.89 ↕ 8.96	37.94 27.85 ⊙ 6.55	40.72 36.13 ↔ 11.71	41.53 44.13 ↕ 13.10	40.61 31.85 ⊙ 8.27	38.98 32.47 ↔ 9.65	36.20 36.13 ↕ 12.41
37.59 31.85 ⊙ 9.65	35.04 28.82 ↔ 8.27	33.30 32.75 ↕ 11.03	36.20 24.80 ⊙ 6.34	38.17 37.36 ↔ 8.96	43.27 47.16 ↕ 14.48	39.56 33.37 ○ 8.27	38.05 23.86 ↔ 6.83

South Side

*Tests performed by NASA/ARC

**2% deflection

Strong (above 4.04×10^5 n/m^2)

Density kg/m^3

Strength N/m^2 x 10^{-4}

Direction Modulus N/m^3 x 10^{-6} **

TABLE 2-16. MECHANICAL PROPERTIES OF 108 E-GLASS/SURFACE MAT/PBI

Thermal Exposure Temperature °K (°F)	Tensile Strength N/m^2 (psi)	Modulus N/m^2 (psi)	Strain to Failure (percent)
RT	7.38×10^7 (10,700)	2.41×10^9 (3.5×10^5)	3.6
422 (300)	5.52×10^7 (8,000)	2.07×10^9 (3.0×10^5)	3.1
542 (515)	4.08×10^7 (5,920)	1.50×10^9 (2.17×10^5)	3.3

TABLE 2-17. MECHANICAL PROPERTIES OF SURFACE MAT/PBI SKIN

Thermal Exposure Temperature °K (°F)	Tensile Strength N/m^2 (psi)	Modulus N/m^2 (psi)	Strain to Failure (percent)
RT	1.95×10^7 (2830)	1.01×10^9 (1.46×10^5)	2.3
533 (500)	1.15×10^7 (1670)	8.62×10^8 (1.25×10^5)	1.7

2.4.2 Thermophysical Properties

Thermal expansion measurements were made on the PBI foam as the skins used in packaging the foam. These measurements, along with the tensile strength measurements, provide the required information for performing thermostructural analysis of the tensile concept.

Between RT and 394°K (250°F), measurements to obtain the PBI skin expansion coefficients showed no net expansion. This was undoubtedly due to the initial loss of water and shrinking of the PBI skins. Subsequently, thermal expansion measurements of the skins were obtained separating the thermal and humidity effects using the DuPont Thermal Analyzer. The results of these measurements for the various materials used in the PBI tile were as follows:

108 E-glass/surface mat/PBI @ 54 percent resin –
548°K - 648°K (526°F - 706°F); α = 16.2 x 10^{-6}m/m°K

Surface mat/PBI @ 76.5 percent resin –
323°K - 673°K (121°F - 751°F); α = 23.2 x 10^{-6} m/m-°K

PBI (64.1 kg/m^3 (4 pcf)) –
473°K - 546°K (391°F - 523°F); α = 54 x 10^{-6} m/m-°K

Aluminum calibration –
400°K (260°F); α = 24.9 x 10^{-6} m/m-°K

The measurements taken on the PBI skin fabrications were taken edgewise on composites fabricated from layers of the skin material. These measurements may not accurately reflect the expansion coefficient of a single ply. However, they appear to be adequate for the initial design screening/evaluation.

Two PBI foam specimens with nominal densities of 56.1 and 72.1 kg/m^3 (3.5 and 4.5 lb/ft^3) were forwarded to Dynatch for thermal conductivity measurements. The materials were tested in accordance with ASTM C177, "Thermal Conductivity of Materials by Means of the Guarded Hot Plate" with the system enclosed in a bell jar evacuated to 0.0133 N/m^2 (1 x 10^{-4} torr). Table 2-18 presents the results obtained in this test series.

Thermal conductivity measurements were previously made (prior to this program) on similarly processed PBI foam with a nominal density of 80 KG/m^3 (5 lb/ft^3). Arc testing by both Aerotherm and NASA as well as additional guarded hot plate measurements were performed to obtain these data. Figure 2-4 compares these data with those described in Table 2-18. Both nominal and upper bound conductivities were defined for subsequent analyses based upon this presentation of available data. At elevated temperatures guarded hot plate measurements on low density insulators become inaccurate. Hence, the data reduced from arc testing was

TABLE 2-18. THERMAL CONDUCTIVITY OF PBI FOAM

Temperature		Thermal Conductivity			
		ρ = 56.1 kg/m^3 (3.5 pcf)		ρ = 72.1 kg/m^3 (4.5 pcf)	
°K	°F	$\frac{W}{m\text{-}°K}$	$\frac{Btu}{ft\text{-}hr\text{-}°F}$	$\frac{W}{m\text{-}°K}$	$\frac{Btu}{ft\text{-}hr\text{-}°F}$
294	70	0.014	0.0081	0.016	0.0092
505	450	0.049	0.0283	--	--
672	750	0.120	0.0690	0.120	0.0690

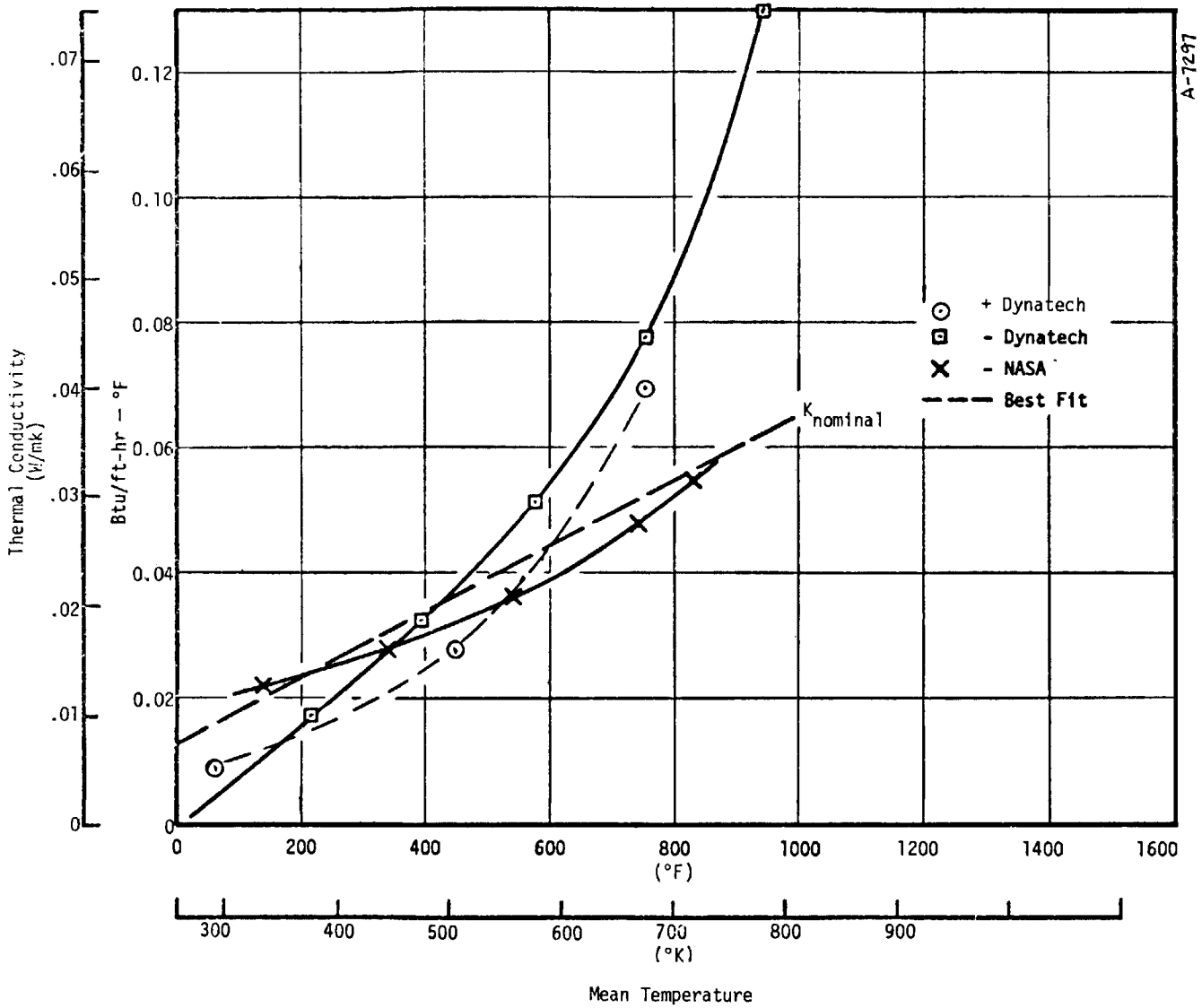


Figure 2-4. Thermal conductivity of PBI foam.

weighted heavily at high temperatures in defining reasonable nominal and upper bound thermal conductivity values for use in subsequent analyses.

2.5 TASK 4 – SYSTEMS ANALYSIS

Sensitivity analyses, thermal sizing, and structural evaluations of the baseline PBI tile concept were conducted to identify the optimum PBI tile construction, attachment techniques, and size. The sensitivity analyses were performed to determine the potential tradeoffs available in selecting various coating systems as well the effects of bondline thicknesses and foam densities upon system performance. Subsequently, the tile thermal and structural sizing constraints were evaluated for typical shuttle upper surface entry environments (represented by the 617°K, 728°K, and 811°K (650°F, 850°F, and 1000°F) isotherms). The basic modeling assumptions used in the sensitivity study were:

- Heating profiles were from space shuttle trajectory 140-40
- Initial temperature = 294°K (70°F)
- Maximum bondline temperature = 450°K (350°F)
- The nominal material properties are:

  – PBI

  ρ = 64 kg/m^3 (4 lb/ft^3), C_p = 1464 J/kg-°K (0.35 Btu/lb-°F)

Temperature, °K(°R)	Thermal Conductivity W/m-°K (Btu/ft-sec-°F)
256 (460)	0.0172 (0.00000277)
311 (560)	0.0311 (0.000005)
811 (1460)	0.1127 (0.0000181)

  – RTV

  ρ = 1409 kg/m^3 (88 lb/ft^3), C_p = 1464 J/kg-°K (0.35 Btu/lb-°F)

  Thermal conductivity = 0.3288 w/m°K (0.0000528 Btu/ft-sec-°F)

  – Al

  ρ = 2690 kg/m^3 (168 lb/ft^3), C_p = 837 J/kg-°K (0.2 Btu/lb-°F)

  Thermal conductivity = 17.25 W/m -°K (0.00277 Btu/ft-sec-°F)

- The nominal coating is described by:
  - Coating weight (including skins and VHT coating) = 0.4496 kg/m^2 (0.092 lb/ft^2)
  - Surface emittance = 0.8

The sensitivity of the TPS sizing to variations in coating properties, PBI thermophysical properties, and bondline thickness are presented in Figures 2-5 through 2-9. In these sensitivity results, the unit system weight is composed of:

- Nominal coating weight
- PBI foam weight
- Bond weight

The coating's optical properties have a significant impact upon the resultant TPS section weight. As shown in Figure 2-5, a variation in the surface emittance from the nominal value of 0.8 down to 0.6 results in approximately a 15 percent increase in the unit system weight. This sensitivity has been presented as a function of the peak heating rate representative of typical shuttle upper surface heating profiles. By comparison, approximately a 6 percent variation in system weight results when the bond thickness is increased 67 percent from the nominal value (reference Figure 2-6). Observing Figure 2-7, one may conclude that the TPS unit system weight exhibits a strong sensitivity to the aluminum substrate thickness upon which the TPS is mounted.

Typically, foam thermal conductivity varies with density as well as temperature. To assess the importance of this effect, the PBI thermal conductivity was assumed to exhibit the same density dependence as a typical foam. The results presented in Figure 2-8 indicate that lower density foam produces lower system weights. However, a 3 kg/m^3 (1/2 lb/ft^3) density variation only reduces the system weight by approximately 9 percent. The effect of maximum surface temperature upon the system weight required to limit the bondline temperature to 450°K (350°F) is demonstrated in Figure 2-9. Thermal analyses have indicated that the coating weight does not influence the system performance due to its surface thermal capacitance effects. Consequently, the coating weight is simply treated as an additive term to the unit system weight. Additional coating development work performed throughout the program does not change the thermal analysis results presented except from the standpoint of optical property variations.

These initial sensitivity studies were performed early in the program to give direction to the subsequent tile development effort. When it was determined that the baseline thermal control coating had better optical properties with approximately the target weight (0.425 kg/m^2 (0.087 lb/ft^2) actual vs. 0.449 kg/m^2 (0.092 lb/ft^2) target) several additional thermal sensitivity analyses were performed. Figure 2-10 displays the effect of improved surface emittance upon the system weight [(0.425 kg/m^2 (0.087 lb/ft^2)(coating + skins), 0.215

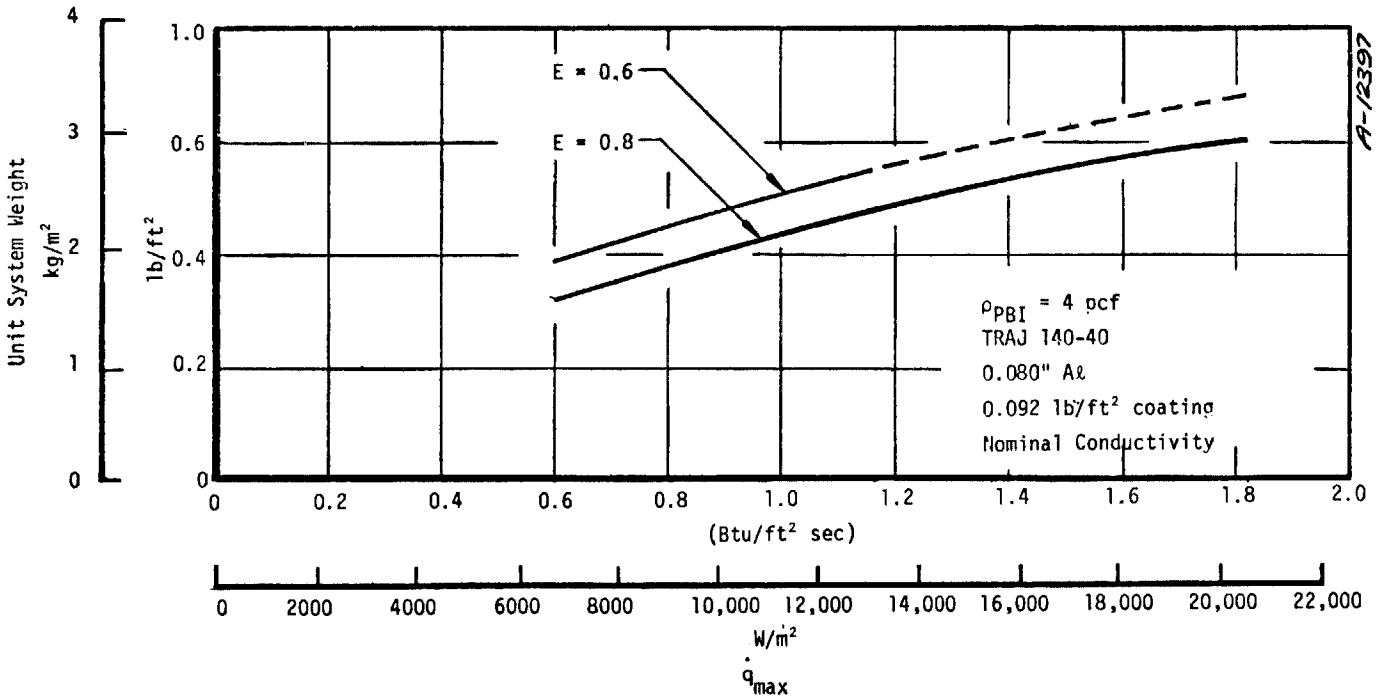


Figure 2-5. System weight sensitivity to surface optical properties.

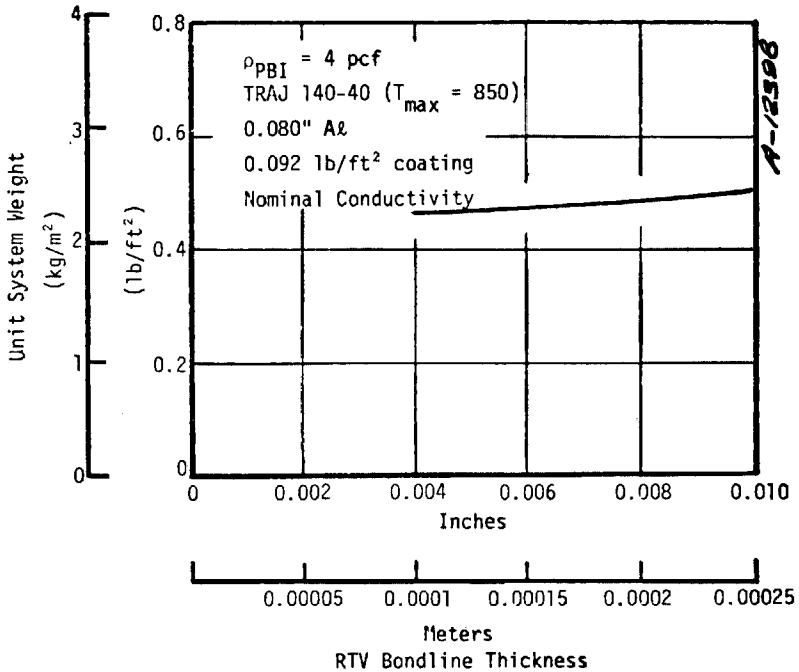


Figure 2-6. System weight sensitivity to RTV bondline thickness.

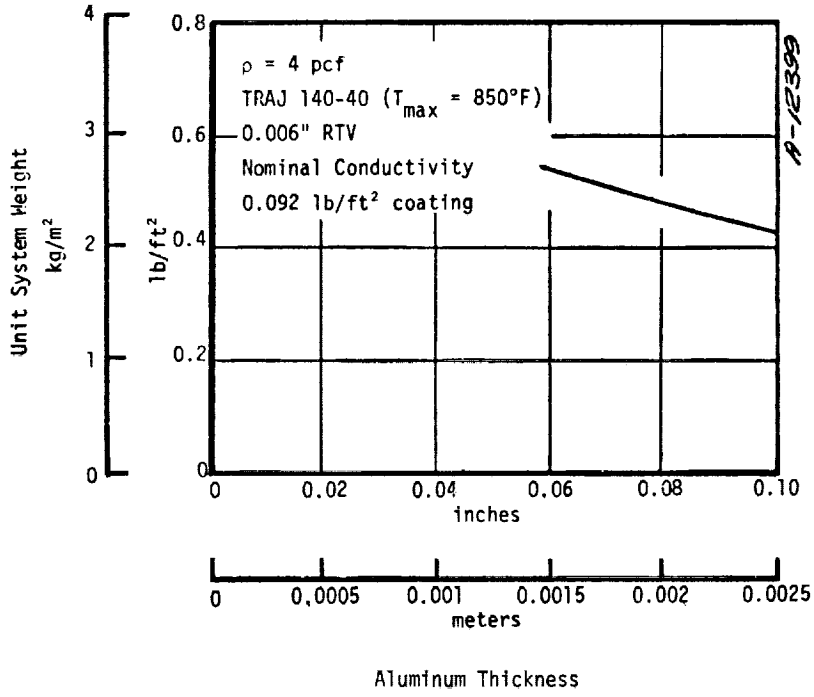


Figure 2-7. System weight sensitivity to aluminum substrate thickness.

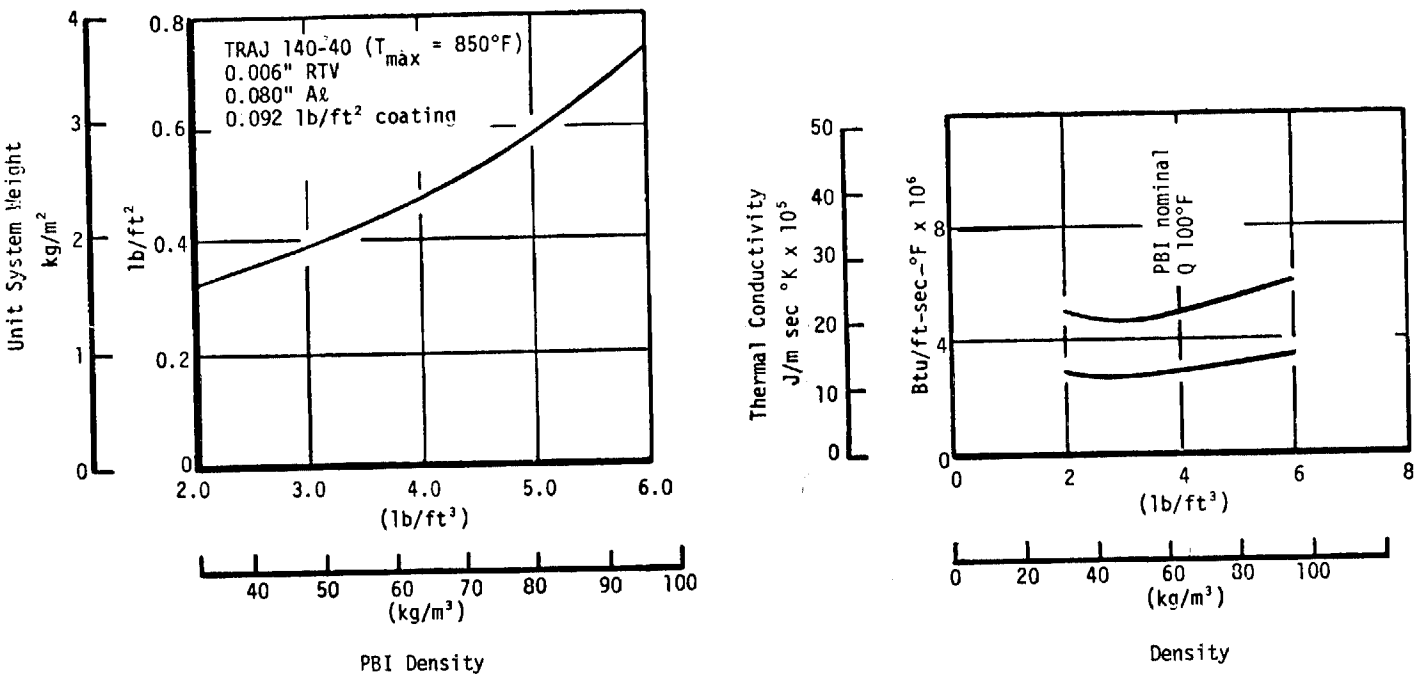


Figure 2-8. System weight sensitivity to PBI foam density.

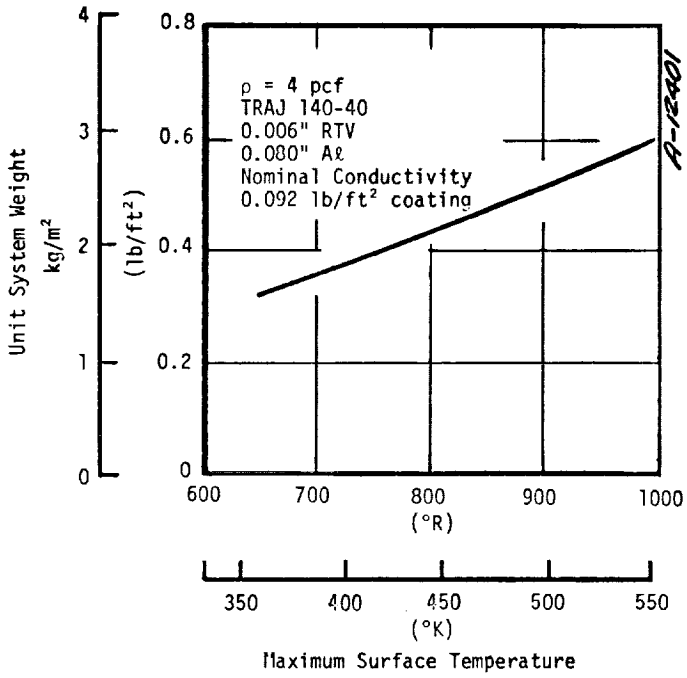


Figure 2-9. System weight sensitivity to maximum surface temperature.

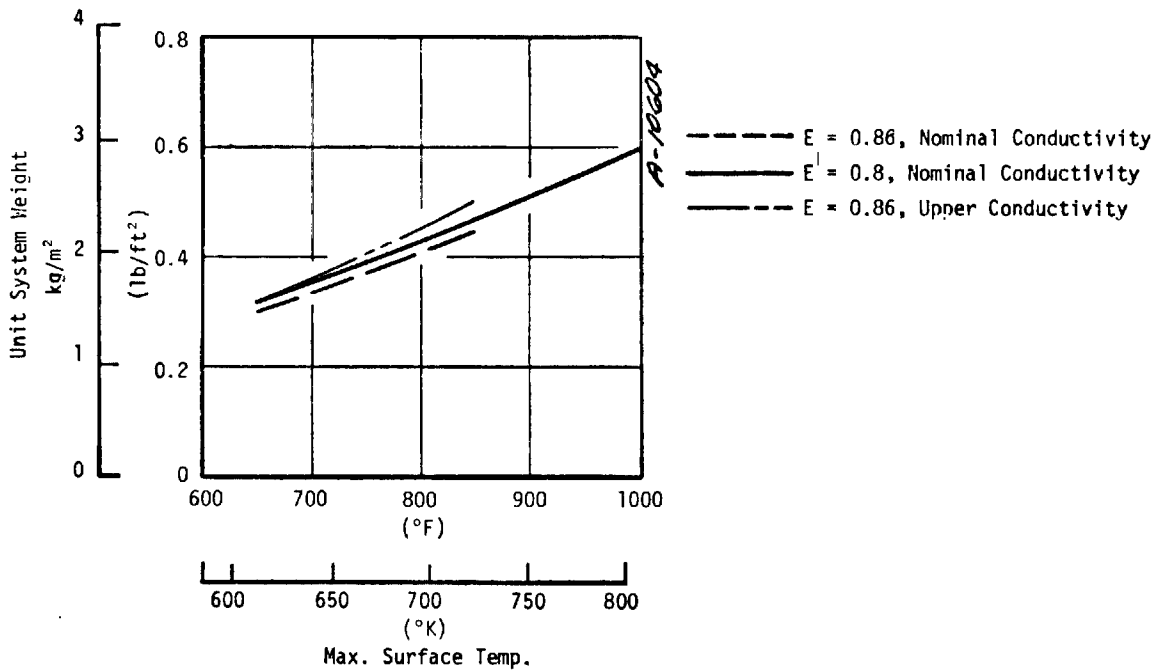


Figure 2-10. PBI system weight predictions.

kg/m^2 (0.044 lb/ft^2) RTV-560 bond, and PBI foam weight @ 64 kg/m^3 (4 lb/ft^3)]. The effects of using various interpretations of PBI thermal conductivity are also depicted. These thermal conductivity values (upper and nominal) were described in Section 2.5.2.

A comparison between the current baseline system weight and the reference system weight at the outset of the program (Table 2-19) shows that the most significant weight savings has been achieved in the coating and surface skin components of the tile.

2.5.1 Thermostructural Sizing

Simplified structural analyses were performed to evaluate the integrity of the PBI tile concept under thermal loading typical of shuttle upper surface environments. Several screening tests were performed and evaluated analytically to verify the analytical techniques and material properties to be used in the tile thermostructural sizing. The objectives of the tile stress analyses were to identify critical tile structural elements, potential failure modes, and maximum tile size.

Various 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") tile layups were evaluated experimentally and analytically in a series of screening tests, and these layup configurations are summarized in Table 2-20. Classical plate analysis techniques (Reference 6) were employed in the analyses performed.

Analyses quite clearly indicate that the material properties strongly affect the predicted deflection. It has been implicitly assumed that within small deflection theory, the predicted deflections should be quite accurate. Consequently, deviations were attributed to material property uncertainties with the ranking of materials relative to confidence in the material properties shown below:

1	Aluminum	High Confidence
2	PBI	↓
3	RTV	↓
4	E-glass	↓
5	Mat	Low Confidence

Of the five PBI tile material components, aluminum, PBI, and RTV material properties are well documented. Thus, in the analysis performed, only the E-glass and surface mat properties were adjusted in an attempt to match the test data.

TABLE 2-19. COMPARISON OF REFERENCE AND CURRENT PBI LRSI TILE (WEIGHT COMPARISON)

Component	Reference System Weight kg x 10^3 (lbs)	Current Baseline System Weight kg x 10^3 (lbs)
Coating (top & sides)	9.7 (0.0214)	~2 (0.0044)
Surface skins	16.6 (0.0366)	~8.0
Foam (4 pcf)	18.91 (0.0417)	18.91 (0.0417)
RTV bond	6.6 (0.0146)	~5.0 (0.0110)
Total system weight (excluding structure)	51.81 (0.1143) 2.25 kg/m^2 (0.46 lb/ft^2)	33.9 (0.0747) 1.46 kg/m^2 (0.30 lb/ft^2)

- For a 0.152 m x 0.152 m x 0.0127 m (6" x 6" x 1/2") tile

TABLE 2-20. THERMOSTRUCTURAL SCREENING SPECIMEN CONFIGURATIONS

Specimen Number	Configuration
1	PBI
2	Mat/RTV/Al
3	E-Glass/Mat/PBI/Mat
4	E-Glass/Mat/PBI/RTV/Al
5	E-Glass/Mat/PBI/Mat/RTV/Al

For the screening tests previously mentioned, a temperature gradient was imposed upon each test specimen (644°K (700°F) front face to 450°K (350°F) backface), and the resultant tile deflections were recorded. The first specimen described in Table 2-20 was tested to verify the PBI properties. Analyses of specimen No. 2 test results provided information on the surface mat used in the layup. Next, the E-glass properties were evaluated throughout tests performed on specimen No. 3 of the screening test series. Subsequent screening tests were evaluated to confirm the results indicated by the first three tests. The material properties resulting from these analyses are summarized in Table 2-21.

Some discrepancies in modulus and expansion coefficient values resulted for both the E-glass and surface mat materials, but it is presently felt that these variations are most likely within the testing uncertainties.

Thermostructural analyses of the screening tests indicated that within the linear plate theory, the deflection predictions (summarized in Table 2-22) are quite good. Negative deflections indicate a convex heated surface. In all cases, the direction of curvature is correctly predicted. Finally, the most accurate predictions are for tests which most closely correspond to the flight configuration (tests numbers 4 and 5 from Table 2-20).

Thermostructural analyses of the baseline PBI tile were performed for a typical shuttle upper surface environment at the time of peak thermal gradient through the tile. Transient thermal response predictions were performed with the Charring Material Thermal Response and Ablation (CMA) program (Reference 7) for two tile configurations:

- 0.152 m x 0.152 m x 0.0140 m (6" x 6" x 0.55") tile with 617°K (650°F) maximum surface temperature
- 0.152 m x 0.152 m x 0.0140 m (6" x 6" x 0.95") tile with 728°K (850°F) maximum surface temperature

TABLE 2-21. MATERIAL PROPERTIES FOR THERMOSTRUCTURAL ANALYSES AT 450°K (350°F)

Material	Modulus		α		ν
	N/m^2	psi	m/m-°K	in/in-°F	
Aluminum	4.04×10^{14}	8.5×10^{6}	2.53×10^{-5}	14.05×10^{-6}	0.33
PBI	1.85×10^{11}	2.9×10^{3}	9.72×10^{-5}	$54. \times 10^{-6}$	0.30
RTV	2.28×10^{8}	4.8×10^{2}	1.575×10^{-4}	87.5×10^{-6}	0.50
E-Glass	4.75×10^{13}	10.0×10^{5}	7.20×10^{-6}	4.0×10^{-6}	0.30
Surface Mat	9.51×10^{12}	2.0×10^{5}	1.62×10^{-5}	9.0×10^{-6}	0.10

TABLE 2-22. SCREENING TEST RESULTS

Specimen No.	Test Deflection		Analysis Deflection		Limit for Plate Theory	
	(m)	(in.)	(m)	(in.)	(m)	(in.)
1	-0.00856	-0.3125	-0.00518	-0.1890	-0.00137	-0.050
2	0.00055 → 0.00082	0.02 → 0.03	0.000214	0.0078	0.000208	0.0076
3	-0.00171	-0.0625	-0.000184	-0.0067	-0.00142	-0.052
4	0.00082 → 0.0011	0.03 → 0.04	0.000876	0.0320	0.00159	0.058
5	0.00137	0.05	0.000899	0.0328	0.00162	0.059

The peak thermal gradient, as deduced from the CMA transient thermal response computations, was used in the stress calculation for the PBI tile; the results of the tile structural analyses are summarized in Table 2-23. For the 728°K (850°F) maximum surface temperature case, the most critical component to the tile survival is the E-glass. Large margins of safety are observed for all the other tile components in both cases. Subsequently, stress calculations were performed to estimate the maximum tile dimensions that could be used in the case of a 728°K (850°F) peak surface temperature exposure. These results indicate that the E-glass layer limits the tile size to approximately a 0.203 m (8" x 8") cross section.

It is concluded that the baseline PBI tile concept is structurally adequate within the tile use temperature range (117°K - 728°K (-250°F to 850°F)) for sizes up to approximately 0.203 m x 0.203 m x 0.0254 m (8" x 8" x 1").

2.6 Quality Assurance

A review of quality assurance program requirements for the production of coated PBI tiles was conducted to insure NASA that continuity could be maintained if the need should arise to scale-up the PBI development to a full scale production effort. Inspection of documentation, identification and evaluation of quality problems, and maintaining overall liaison with WR&D quality assurance personnel were accomplished under this effort. Recommendations for a comprehensive quality control program were made.

For flight applications of PBI tiles, a basic quality assurance system that complies with the requirements of NASA NHB 5300-4(1B) "Quality Program Provisions for Aeronautical and Space System Contractors" must be employed.

The quality assurance organization must assure that all projects and products meet acceptable standards of quality. Complete documentation must be maintained on pertinent attributes and measured values on raw materials, processing, and completed tile fabrications. These records must provide acceptable tracability for each article shipped. There must be, in addition, a program for periodically verifying the accuracy of all measuring instruments and devices, and for calibrating all processing equipment.

Utilizing the requirements contained in the material and process specifications, Quality Assurance must review and approve all purchase orders prior to their issuance. Incoming materials must be inspected for compliance to the purshase order and applicable specifications. Prior to storage, the accepted materials must be suitably stamped and material acceptance must be verified by an inspector prior to fabrication.

TABLE 2-23. PBI TILE THERMOSTRUCTURAL CALCULATIONS

Maximum Surface Temperature °K (°F)	Tile Thickness m (in.)	Tile Component	Calculated Stress N/m^2 (psi)	Allowable Stress N/m^2 (psi)
617 (650)	0.0140 (0.55)	Aluminum	1.49×10^6/-8.23×10^6 (216 max/-1193 min)	--
				--
		RTV-560	-1.25×10^5 (-18.2)	1.36×10^6 (198)
		Surface Mat	2.05×10^6 (297.2)	7.79×10^6 (1130.)
		PBI Foam	5.63×10^4 (8.16)	2.76×10^5 (39.96)
		Surface Mat	3.03×10^6 (440)	7.80×10^6 (1131)
		E-Glass	2.86×10^6 (415)	4.00×10^7 (5800)
723 (850)	0.0241 (0.95)	Aluminum	540×10^6/-1.23×10^7 (783 max/-1784 min)	--
				--
		RTV-560	-7.10×10^4 (-10.3)	1.36×10^6 (198)
		Surface Mat	1.27×10^6 (184.6)	7.76×10^6 (1126.)
		PBI Foam	8.07×10^4 (11.7)	2.76×10^5 (40.)
		Surface Mat	4.34×10^6 (629.7)	7.75×10^6 (1124)
		E-Glass	2.40×10^7 (3487.7)	3.70×10^7 (5369)

A complete fabrication process including all material inputs should be formulated including inspection points to assure that all technical and workmanship requirements are met. This provides documentation of compliance to all requirements for each tile or batch of tiles fabricated.

In the event that materials, process, or tile assemblies do not conform to established requirements, a review procedure should be initiated. While the review is in progress, the parts under consideration should be withdrawn from production and controlled by Quality Assurance. Records of such actions should be maintained as they provide a basis for performance review and corrective actions when warranted.

Production quality control should be monitored continuously with nondestructive testing (NDT) techniques to provide monitoring of the quality of materials and to assure the maintenance of the PBI tile performance within given design constraints. X-ray techniques can be used to measure the nonuniformity of the PBI foam and identify large voids, cracks, or other discontinuities. This technique will not necessarily provide a measure or an assessment of the effect of these defects upon the foam's thermal performance, however, it provides a low cost method for obtaining a first order assessment of the foam quality.

Infrared testing could be effectively employed as a screening test for PBI foam and PBI tiles. When a thermal gradient is established across the foam, nonuniformities which affect the insulative qualities of the product can be recorded with IR film or scanning temperature sensors. This IR technique could also be used to detect thermal inconsistencies in either the skins or the thermal control coating.

From each processed "batch" of PBI foam, various destructive testing should be performed on samples to assure consistency of the mechanical properties of the PBI foam. Tensile and compression tests should be performed both after production and after thermal aging to establish baseline properties and property retention characteristics of the foam.

Tile inspections would reject tiles not conforming to design dimensional (length, width, flatness, etc.) or weight constraints. All inspection and test data should be documented to establish traceability and historical data.

By implementing the above mentioned quality assurance measures, it is felt that NASA can be assured of reproducability for the coated PBI tiles in a full production program.

SECTION 3

CONCLUSIONS AND RECOMMENDATIONS

An improved PBI coated tile TPS which satisfied all the program objectives was developed in this effort. Both material and processing specification for the PBI foam, and a material specifications for the selected coating system were prepared.

PBI packaging techniques were investigated due to requirements:

- To provide dimensional stability to the PBI foam
- To provide a continuous smooth, nonporous bondable surface which acts as a substrate for the thermal control coating
- To provide the foam with a barrier to oxidative attack
- To provide a tough surface which will resist mechanical damage during handling and will resist erosion of the face and edges of the tile during flight

The no overlay concept, where the PBI impregnated skins are cured directly to the foam, was identified as the optimum packaging concept from both weight and ease of fabrication standpoints. Several fabrication problems identified and solved through the foam packaging development efforts were:

- Tile weight control
- Side surface flatness
- Tile warpage
- Surface dimpling and porosity

Alterations of the processing techniques provided solutions in all cases. Controls on the PBI preimpregnation of the skins provided the processing control required on the tile weight. Side surface flatness and tile warpage were controlled by mold modifications and variations on processing techniques. Surface dimpling and porosity were eliminated by appropriately selecting combinations of skin materials.

Potential thermal control coatings evaluated in the initial screening phases included:

Flame sprayed – ZrO_2, Al_2O_3

Sprayable – VHT SP 101 (TiO_2 filled silicone)

– DC 805 TiO_2 filled silicone elastomer

– Sauereisen ceramics

These candidate coatings were screened to determine if they could satisfy the 100 mission reuse, ultraviolet exposure, optical property, temperature limit, and handlability design constraints typical of the shuttle upper surface. Fabrication evaluations and screening tests were the basis for the selection of the optimum thermal control coating. Flame sprayed Zirconia abraided the PBI skins during application, and consequently was an unsuitable coating. The flame sprayed alumina coating was extremely heavy and difficult to fabricate and hence was not considered to be a viable candidate. The sauereisen coatings did not bond well to the PBI skins, and hence were not considered acceptable. VHT SP 101 and the TiO_2 loaded DC 805 (2 parts TiO_2) both have acceptable optical properties, however, thermal stability testing indicated that the DC 805 coating was significantly more brittle after heat soak at 650°F, and hence VHT SP101 was selected as the prime coating system.

PBI foam has a residual moisture content of approximately 4 percent. An evaluation was performed to determine if treatment of the foam with known hydrophobic compounds could reduce this residual moisture content. After treatment, the foam moisture content was unchanged while the foam weight was increased by the amount of hydrophobic compound absorbed. Treatment of the tile with DC 530 however provided a waterproofing effect which suggests that hydrophobic treatment of the tile will provide some rain resistance for a system weight increase of from approximately 5 to 7 percent.

Initial radiant lamp screening tests performed by NASA/ARC have shown the developed tile to be thermally stable up to 728°K in a vacuum. The tile also has excellent impact resistance characteristics. Adhesive evaluations resulted in the selection of RTV 560 as the bonding material to attach the tile to an aluminum structure.

Systems analyses were performed to identify the system weight sensitivity in a typical shuttle environment to design variables including foam density, bondline thickness, and the optical properties of the thermal control coating. Bondline thickness changes of 67 percent only have a 6 percent effect on the system weight, while a 12 percent change in foam density results in approximately a 9 percent change in the computed system weight.

Optical properties significantly affect the PBI TPS weight. A change in surface emittance from 0.8 to 0.6 alters the system weight requirement by 15 percent.

Thermostructural analyses of the baseline tile concept indicated that at a surface temperature of 728°K (850°F), the most critical component to the tile survival is the E-glass skin. Preliminary analyses suggest that an E-glass structural failure limits the tile cross section to approximately 0.203 m x 0.203 m (8" x 8") at this peak surface temperature level.

The developed PBI TPS weight was compared to the silica RSI TPS proposed for the shuttle upper surface. Based upon a minimum gage system, which is typical in areas with peak surface temperatures of less than 617°K (650°F), a weight savings of 0.17 lb/ft^2 could be realized by using the PBI insulation system. This savings is primarily attributable to the elimination of the strain isolation pad which is required for the RSI TPS. Hence, for shuttle applications, a vehicle weight savings of approximately 600 lbs could be realized if PBI were to replace RSI in only those areas with peak surface temperatures less than 617°K (650°F). Additional weight savings potential over the RSI system exists up to the PBI reuse temperature limitations of 728°K (850°F), and additional fabrication refinements could further increase the weight savings.

The coated PBI tiles developed under this effort offer a low weight, thermally efficient, rugged, stable, thermal protection system for reuse applications where insulation temperatures do not exceed 728°K (850°F). The utility of applying this versatile insulation system in thermally and mechanically severe environments should be investigated further.

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3. Tang, S., "Thermal Stresses in Temperature-Dependent Isotropic Plates," Journal of Spacecraft and Rockets, Vol. 5, No. 8, August 1968, p. 987.

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APPENDIX A

MATERIAL AND PROCESSING SPECIFICATION FOR POLYBENZIMIDAZOLE FOAM

A.1 SCOPE

This specification establishes the processing requirements and new materials necessary to produce a rigid polybenzimidazole foam insulation.

A.2 DESCRIPTION

The rigid PBI foam billets produced by this specification yields a foam which does not exhibit melting behavior up to 1089°K (1500°F) and yet has excellent toughness as demonstrated by its tensile elongation of ~5 percent at room temperature.

A.3 PROCESS DESCRIPTION

A.3.1 Polybenzimidazole Propolymer

The material shall be a condensation prepolymer based on 3,3',4,3'-tetraaminobiphenyl and diphenyl isophthalate.

A.3.1.1 Raw Material Requirements

A record of lot numbers and incoming inspection results shall be kept on each lot of raw material used for prepolymers used to produce the foam.

A.3.1.1.1 Diphenyl Isophthalate

The diphenyl isophthalate shall be in accordance with the following specifications:

a. Saponification equivalent: 159 ± 1

b. Acid content: 0.10 percent maximum

c. Melting point: 133.0°C minimum

d. Ash content: 0.1 percent maximum

A.3.1.1.2 3,3',4,4'-Tetraaminobiphenyl

The tetraamine shall be in accordance with the following specifications:

a. Amine content (by acetylation): 97 percent minimum

b. Chlorine content: 0.05 percent maximum

c. Moisture content: 0.3 percent maximum

d. Melting point: 173°C minimum

e. Ash content: 0.1 percent maximum

f. Dimer content: irrelevant

A.3.1.2 Prepolymer Quality Control

Form: The material shall be a ground powder, uniform in quality and free of foreign materials. Sieve analyses conducted in accordance with Paragraph A.3.1.2.1.1 shall show a maximum of 0.5 percent retained on a 50 mesh sieve; a maximum of 25 percent retained on a 100 mesh sieve; and a minimum of 13 percent passing through a 200 mesh sieve.

Color: The material shall be light tan in color. The material shall have a maximum color index of 0.40 when tested in accordance with Paragraph A.3.1.2.1.2.

Melting Point: The material shall have an initial melting point of 356°K - 382°K (181°F - 228°F), and a final melting point of 431°K - 445°K (316°F - 341°F) when tested in accordance with Paragraph A.3.1.2.1.3.

Volatile Content: The volatile content shall be 23 to 27 percent when tested in accordance with Paragraph A.3.1.2.1.4.

Insolubles: Insoluble material shall be less than 0.1 percent when tested in accordance with Paragraph A.3.1.2.1.5.

Material which meets the requirements of this specification shall be so identified and released for foam production use.

A.3.1.2.1 Test Procedures

A.3.1.2.1.1 Sieve Analysis

Place 0.1 kg into the top of a stack of full-size [0.203-meter (8-inch) diameter] standard sieves; stacking order from the top shall be: cover, 50-mesh, 100-mesh, 150-mesh,

200-mesh, 250-mesh, pan. Shake for 1 hour on a Cenco-Meinzer Sieve Shaker at a setting of 8. Determine the weight gain of each sieve and pan. Report as percent weight retained by each mesh.

A.3.1.2.1.2 Color Test

Weigh a 67 x 10^{-6} to 100 x 10^{-6} kg sample of 200 mesh PBI prepolymer into a 5 x 10^{-6} m^3 (0.050 l) stoppered flask. Add [0.500 x sample weight (mg)] ml ± 1 ml of spectrograde acetone. Shake vigorously for exactly two minutes. Immediately filter the solution through a coarse filter paper, collecting about 1 x 10^{-5} m^3 (0.010 l) of filtrate.

Dilute 1 x 10^{-6} m^3 (1.00 x 10^{-3} l) of this solution to 1 x 10^{-4} m^3 (0.100 l) with additional acetone.

Determine the adsorbance of the dilute solution of 343 mμ and the adsorbance of the undiluted solution of 435 mμ, using acetone as a reference in each case

$$\text{color index} = \frac{\text{adsorbance of concentrated solution at 435 m}\mu}{\text{adsorbance of dilute solution at 343 m}\mu}$$

A.3.1.2.1.3 Melting Point

The melting point shall be determined, using a standard capillary tube method. The initial, resin, and final melting points shall be determined and recorded.

A.3.1.2.1.4 Volatile Content

The volatile content shall be determined as follows: A 0.0026 ± 0.0003 kg sample in a test tube is heated to 623°K ± 5°K for 720 ± 120 seconds at atmospheric pressure; the sample is held at 623°K ± 5°K for 2 hours under full vacuum. The average percent weight loss of two determinations shall be calculated and the determinations shall exceed a spread of 0.8 percent.

A.1.2.1.5 Insolubles

Weight a 0.020 kg sample of prepolymer into an extracted, dried and weighed 0.033 x 0.094 m soxhlet extractor thimble. Extract the prepolymer in a soxhlet extractor, using pyridine solvent, until the discharge liquid is clear. Continue extraction for an additional 1 hour. Dry the thimble and reweigh.

$$\text{insolubles (percent)} = \left(\frac{\text{weight of residue}}{\text{weight of sample}}\right)(100)$$

A.3.1.3 Packaging of Prepolymer

The material shall be packaged in opaque, sealable containers. Each container shall be marked with the following information:

a. Manufacturer's name

b. Product name and number

c. Batch number

d. Date of manufacture

e. Warranty expiration date

f. Net weight

A.3.2 Foam Preparation

A.3.2.1 Prepolymer Requirements

The polybenzimidazole product meeting the requirements of Paragraph A.3.1.2 shall be used as the base material for foam processing. Quantities of this material to be used in any specific process will be determined by the density and configuration of the required insulation. For instance, if the polymer had a volatile content of 20 percent as determined in Paragraph A.3.1.2.1.4 and a 64.1 kg/m^3 (4.0 pcf) foam is sought in a 0.028 m^3 (1 cubic foot) mold cavity, one would charge the mold with 2.27 kg (5.0 lbs) of PBI prepolymer. The width and length of the foam slab are limited only by oven or mold size but the height of the mold should not exceed 0.0635 m (2.5 inches) (foaming direction).

A.3.2.2 Facilities and Tooling

Two major pieces of equipment and required; an air circulating oven and a special foaming tool. The oven shall include provision for GN_2 application to the foaming tool and must provide a 200°/hour heating rate up to 783°K (950°F). The foaming tool(s) shall be of 20-gauge 0.302 steel. Foaming tool components are selected as a set based on the required foam density and geometry. The nitrogen used for the foaming operations shall not have more than a 100 ppm impurity level (99.9 percent).

A.3.2.2.1 Foaming Tool

A metal mold of desired dimensions can be constructed as diagrammed in Figure A-1. A typical molding fixture consists of the following parts:

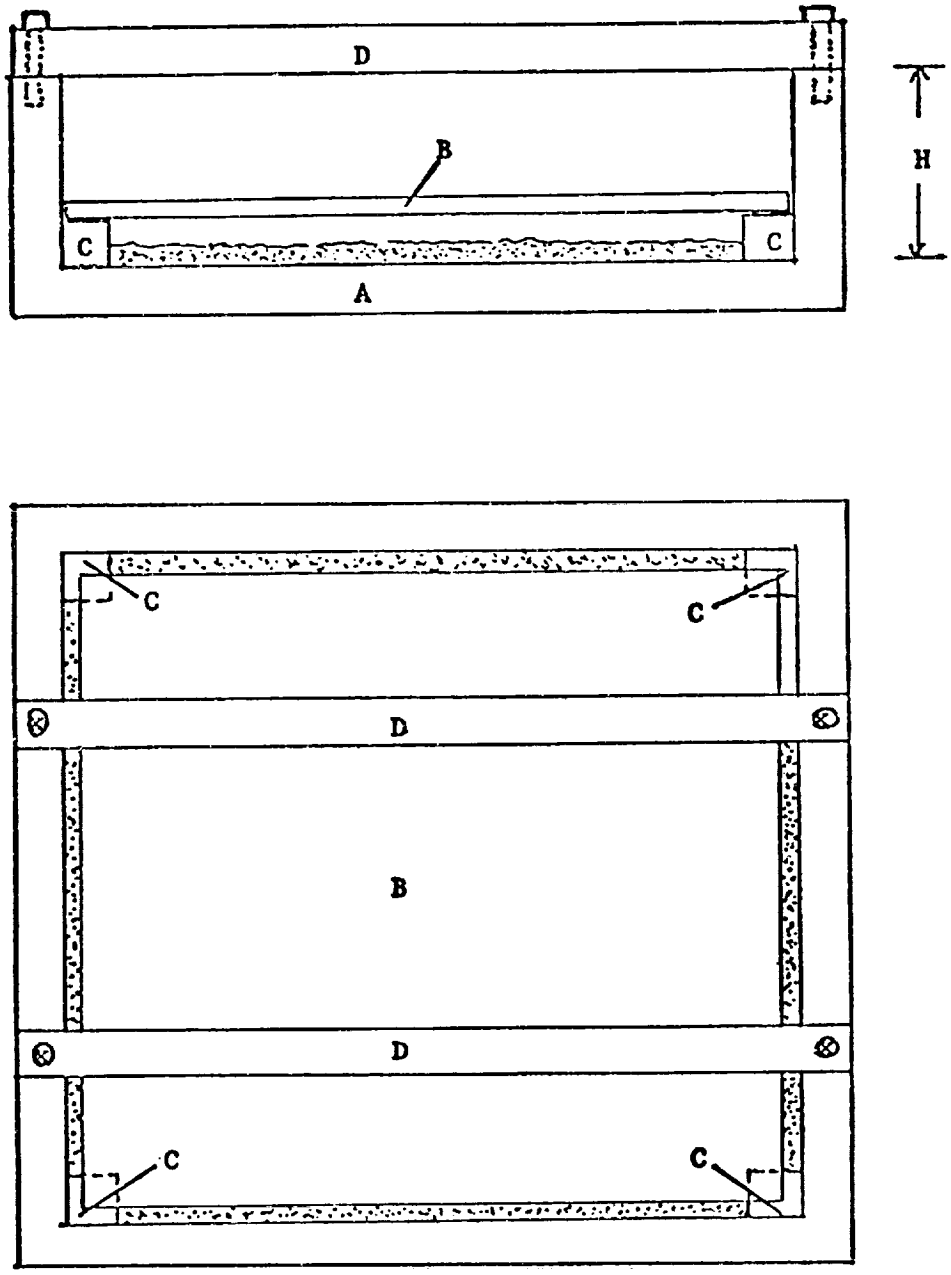

Figure 1

a. Mold cavity (A) is fabricated to suitable dimension which encloses the volume to be filled. The upper molding surface is formed by the lifting action of the rising foam on the lower side of float surface B, which continues until the upper surface of B meets the restraining bars D. The total foam rise then is equal to the total sidewall height H minus the thickness of plate B.

b. The floating plate B serves a twofold purpose: (1) provides a resistance to the rising foam which results in more uniform blowing action, and (2) serves as a secondary heat source which results in a smaller temperature gradient in the rising foam.

c. The plate may be as much as 0.00254 m (0.100 inch) less than the width and length dimension in A – this gap allows release of the majority of the volatiles during the preliminary foaming procedure.

d. Floating plate B is positioned above the prepolymer and supported by stand-offs of suitable dimension so that the lower surface of the plate is not in contact with either the solid or melted prepolymer until the foaming has proceeded to ~20 percent of the final volume.

e. Restraint bars D are bolted in place and the entire fixture is then placed in a forced air oven.

A.3.2.3 Processing

A.3.2.3.1

The appropriate amount of prepolymer (reference Paragraph 3.2.1) is evenly distributed inside the selected foaming tool housing. Leveling plate supports are installed and the leveling plate adjusted before the housing cover is sealed.

A.3.2.3.2

The complete foaming tool with charge is placed in an oven at room temperature and a GN_2 purge system is connected. A temperature sensor located at the center base of the foaming tool is used to monitor the process.

A.3.2.3.3 Curing Schedule

a. Heat to 450°K (350°K) and hold 90 min.

b. Increase temperature to 478°K (400°F) over 75 min.

c. Increase temperature to 533°K (500°F) over 50 min.

d. Hold at 533°K (500°F) for 15 min.

e. Increase temperature to 644°K (700°F) over 50 min.

f. Hold at 644°K (700°F) for 15 min.

g. Increase temperature to 741°K (875°F) oven 50 min.

h. Hold at 741°K (875°F) for 30 min.

i. Cool to 339°K (150°F) with purge on before removing foam from tool

A.3.2.3.4 Postcure

Postcure at 769°K (925°F) for 1 hour in GN_2.

A.3.2.4 Quality Control

Thermal Stability: The foam retains 95 percent of its weight in vacuum or N_2 when heated isothermally for 24 hours at 920°F. A microsample removed from a foam billet tested on the DuPont 900 Thermal Analyzer shall not exceed a weight loss (after water loss) of 1.0 percent after 3 hours at 900°F in an inert atmosphere. This test shall be run in duplicate.

Thermo-oxidative Stability: A microsample shall be tested in the TGA mode (air) with the DuPont 900 Thermal Analyzer. After initial loss of water, the intercept of the two slopes shall be to less then 500°C. This technique is shown below.

Density: The density of the billet shall be determined and it must be between 60.9 and 67.3 kg/m^3 (3.8 and 4.2 pcf).

Color: The color of the foam is dark brown.

Foam Cell Structure: The presence of large holes (0.0127 m) in the foam shall be cause for the billet to be rejected.

Mechanical Properties: The foam shall retain at a minimum 50 percent of its compressive strength after 24 hours of aging at 588°K (600°F) in air when tested in accordance with ASTM-D-1621.

APPENDIX B

MATERIAL SPECIFICATION FOR PBI TILE COATINGS

B.1 SCOPE

These specifications establish the raw materials necessary to produce tiles for insulation of the upper surface of shuttle during reentry.

B.2 DESCRIPTION

The white insulative PBI tiles produced by this specification are made to the desired thickness and size to provide the required thermal protection to the aluminum substructure. RTV 560 is the adhesive used for bonding the tiles to the aluminum skin.

B.3 MATERIAL DESCRIPTION

Some processing notes have been added to this material specification for clarification.

B.3.1 PBI Foam

Foam billets are cut 1.6 percent over the designated size and dried for 1 hour at 400°F.

B.3.2 Surface Mat PBI Prepreg

Owens Corning fiberglass 10-mil surface mat (234 finish) is impregnated with PBI prepolymer (described in specification for polybenzimidazole foam). The weight of this prepreg shall be 0.1162 $kg/m^2 \pm 0.0215$ kg/m^2.

The foam billets described in B.3.1 are wrapped with the prepreg and cured for 1 hour at 672°K (750°F).

B.3.3 108E Glass PBI Prepreg

Owens Corning fiberglass 108 E-glass (silane finish) is impregnated with PBI prepolymer. The weight of this prepreg shall be 0.1076 $kg/m^2 \pm 0.0215$ kg/m^2.

The tile prepared in B.3.2 is surfaced with this prepreg and cured for 1 hour at 728°K (850°F).

B.3.3 VHT SP101

After suitable surface treatment of the tile (sanding), the dried tile is sprayed with VHT SP101 available from Sperex Corporation in Gardena, California. The coating weight to be applied to the tile (5 sides) is 0.0733 kg/m^2 (0.015 lbs/ft^2). The minimum weight shall be that weight which covers the top surface of the tile such that the dark PBI cannot be seen and the maximum weight shall be 0.0861 kg/m^2.